

1986

ANNUAL QUALITY ASSURANCE PERFORMANCE REPORT

SECTION 1

APIOs SAMPLES

INORGANIC TRACE CONTAMINANTS SECTION

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G. C. RONAN, DIRECTOR
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Ministry of the Environment

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1986

ANNUAL QUALITY ASSURANCE PERFORMANCE REPORT

SECTION 1

APIOS SAMPLES

INORGANIC TRACE CONTAMINANTS SECTION

D G STURGIS
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Inorganic Trace Contaminants Section
Laboratory Services Branch
Ministry of the Environment

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INORGANIC TRACE CONTAMINANTS SECTION

SUMMARY

I. Introduction

The Inorganic Trace Contaminants Section of the Ministry of the Environment, Laboratory Services Branch is responsible for the analysis of a wide variety of sample types for metals and non-metals. The use of sensitive instrumentation and methodologies appropriate to the sample matrix, combined with quality assurance programs, ensures that the Section is able to maintain a high standard of analytical performance. This performance is monitored through regular internal quality control and assurance programs as well as participation in interlaboratory round-robins. This QA report summarizes the methodologies used for analysis of these samples and the supporting internal quality assurance data.

This report is assembled in sections that reflect the analyses performed on different sample matrices in support of the programs of the Ministry of the Environment. Coincidentally, these divisions also reflect the supervisory responsibilities within the Section.

II. Quality Control and Assurance

The objectives of the quality control and assurance programs are to ensure that all of the components of the analytical process are under control and to ensure immediate detection and correction of unacceptable analytical performance. The program monitors all of the reagents, instrumentation, calibration and recovery components of the analytical system.

A. Quality Control

Quality control of the analytical process takes place at the instrument level and is intended to ensure that the instrumentation is operating according to established criteria. This control function ensures that instrument calibration, standardization, slope and intercept, and instrumental drift meet these criteria.

B. Quality Assurance

Quality assurance of the analytical process takes place after the results have been generated and is intended to ensure that the analytical protocols of sample preparation and digestion have been carried out correctly. This control function ensures that reagent blanks, digested standards, sample duplicates and recovery materials meet established response criteria.

III. Report Format

The report consists of one page method summaries and one page data summaries of blanks, between-run controls and within-run duplicates in formats that are common to all of the parameter/matrix combinations. The method summaries give a brief outline of the sample preparation and measurement procedures. The data summaries consist of annual mean values with standard deviations.

For the within-run duplicates, the data set is subdivided into ranges approximating 0 to 20 %, 20 to 50 % and 50 to 100% of the analytical range. All results for duplicates reported to the data base that are "<" or that have been diluted into the range are excluded from the statistical analysis.

The standard deviations for blanks and between-run controls are calculated using formula I. Formula II is used for the calculations for within-run duplicates.

$$sd = \sqrt{[(\text{sum}x^2 - (\text{sum}x)^2)/n/(n-1)]} \dots\dots I$$

$$sd = \sqrt{(\text{sum}d^2/2n)} \dots\dots II$$

where : x = the individual values; n = the number of events
d = the differences between pairs of duplicates

The data is stored in a personal computer using BMB Manager II files. All data manipulations, reports generated etc, are performed using applications written in Manager Math.

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ITC SECTION ANNUAL QA REPORT 1986

1. APIOS Lovol Filters and Precipitation Samples

1.1 Lovol Filters

Lovol filters consist of 47 mm circles of Whatman 41 filters that are exposed to approximately 20 cubic meters of air per 24 hour sampling period. True sample duplicates cannot be taken since the whole filter is used during analysis. The samples are extracted initially with water for anion analysis and this extract may be split to generate duplicate data. Metals are determined on an acidified portion of the water leach and a separate second acid leach of the filter material.

1.2 Precipitation samples

Precipitation samples are collected in plastic bags and depending upon the collection devices used, may be either wet only, dry only, or wet and dry composites. Analysis is performed on an acidified portion of the liquid sample or on an acid leach of the container. Duplicates consist of taking separate portions of the original liquid or of the acid leach.

The following table summarizes the parameters determined in APIOS samples, the preparation procedure and method of analysis.

TABLE 1.1

| Parameter | Collection Device | Preparation | Analysis |
|-----------|----------------------------------|-------------|----------|
| Metals | Whatman 41 filter | Acid digest | GFAAS |
| Metals | Plastic bags (liquid portion) | Acidified | GFAAS |
| Metals | Plastic bags (acid leach) | Acidified | GFAAS |

1.3 APIOS Quality Assurance

QA samples consist of EPA solutions and blanks (filters). Duplicates are subsamples of the filter extracts or subaliquots of the contents of the collection bags in the case of the precipitation samples. The following table summarizes the QA sample used for the APIOS sample program.

TABLE 1.2

| Sample Designation | Type | Parameter |
|--------------------|------------------------|-----------|
| epal,epah | EPA standard solutions | Metals |
| blk | Whatman 41 filter | Metals |
| Std4,5 | Standard solutions | Metals |

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Aluminum TEST CODE: ALUR SAMPLE TYPE: Lovol filter
UNIT: Waters Unit SUPERVISOR: P. N. Vijan

METHOD CODE: 527AF2

REVISION NO: 1

DATE: Sept 1986

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-one filter
Container-stored in a plastic petri dish
Preservative-
Other-

SAMPLE PREPARATION: Partial Extn.-yes Total Extn.- % Extracted-

Procedure-The exposed lovol filter is extracted with 50 ml of double distilled water in an ultrasonic bath for 20 min. Twenty ml. of the water extract is taken for anion & cation analysis. The filter and the water extract are separated by decanting solution into separate tube. The water solution is preserved with 1% ultrapure HNO₃. The filter and insoluble contents are further extracted in 50 ml of .16N HNO₃ in a closed tube overnight at 90°C. Both solutions are analyzed by GFAAS. The results from the two solutions are combined in reporting in the units of ug/filter.

20 µL of sample is injected into the furnace and 0 lpm is the internal argon flow at atomization.

INTERFERENCES: Unknown

REPORTING RESULTS: ug per filter

INSTRUMENTATION: Perkin Elmer 603 or 2380 Atomic Absorption Spectrophotometer with a HGA400 or HGA500 furnace and AS40 autosampler

Calibration Range: 0 to .100 mg/L

Resolution: 0.001 Abs. Units

Sensitivity: .030 mg/L = .200 Abs Units

Instrument Detection Limit: .005 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-0 to 10 µg/filter (µg/Fi)

Accuracy-

Precision of Controls-

| | A | B |
|-----------|-------|-------|
| mean | .74 | 7.77 |
| std. dev. | .108 | .702 |
| R.S.D. | 14.6% | 9.03% |

Precision of Duplicates-low range mid range high range

| | | |
|------|------|------|
| s.d. | .050 | .28 |
| mean | 1.10 | 3.50 |

W .5 µg/Fi

T 2.5 µg/Fi

CONTROL LIMITS: The epa controls should be within 15% of the given value

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

ALUMINUM-GF IN LOVOL FILTERS

Operating Range = .50000to 10.000 ug/filter

IN - RUN DUPLICATES

| | | | | | |
|-------|---------|----------------|----------------|----------------|---------|
| Range | <.50000 | .50000to2.0000 | 2.0000to5.0000 | 5.0000to10.000 | >10.000 |
| no. | 6 | 4 | 1 | 0 | 4 |
| s.w. | | 0.05000 | 0.28000 | 0.00000 | |
| mean | | 1.1000 | 3.5000 | 0.0000 | |

QA CONTROL SAMPLES

| SAMPLE I.D. | NO. | MEAN | STD. DEV. | R.S.D. |
|-------------|-----|---------|-----------|--------|
| epal | 54 | 0.74000 | 0.10800 | 14.59 |
| epah | 63 | 7.7700 | 0.70200 | 9.03 |

BLANKS

| BLANK I.D. | NO. | MEAN | STD. DEV. |
|------------|-----|--------|-----------|
| blk | 46 | .84000 | .65400 |

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Cadmium TEST CODE: CDUR SAMPLE TYPE: Lovol filter
UNIT: Waters Unit SUPERVISOR: P. N. Vijan

METHOD CODE: 527AF2

REVISION NO: 1

DATE: Sept 1986

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-one filter
Container-stored in a plastic petri dish
Preservative-
Other-

SAMPLE PREPARATION: Partial Extn.-yes Total Extn.- % Extracted-
Procedure-The exposed lovol filter is extracted with 50 ml of double distilled water in an ultrasonic bath for 20 min. Twenty ml. of the water extract is taken for anion & cation analysis. The filter and the water extract are separated by decanting solution into separate tube. The water solution is preserved with 1% ultrapure HNO₃. The filter and insoluble contents are further extracted in 50 ml of .16N HNO₃ in a closed tube overnight at 90°C. Both solutions are analyzed by GFAAS. The results from the two solutions are combined in reporting in the units of ug/filter.
90 µL of sample is injected into the furnace and 50 lpm is the inter. argon flow at atomization.
INTERFERENCES: Unknown

REPORTING RESULTS: ug per filter

INSTRUMENTATION: Perkin Elmer 603 or 2380 Atomic Absorption Spectrophotometer with a HGA400 or HGA500 furnace and AS40 autosampler

Calibration Range: 0 to .01 mg/L

Resolution: 0.001 Abs. Units

Sensitivity: .005 mg/L = .450 Abs Units- Peak Height Mode

Instrument Detection Limit: .00002 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-0 to 1 µg/filter (µg/Fi)

Accuracy-

Precision of Controls-

| | A | B |
|-----------|-------|-------|
| mean | .040 | .355 |
| std. dev. | .0050 | .0325 |
| R.S.D. | 12.5% | 9.15% |

Precision of Duplicates-low range mid range high range

| | | |
|------|-------|-------|
| s.d. | .0040 | .0204 |
| mean | .039 | .219 |

W .002µg/Fi

T .010µg/Fi

CONTROL LIMITS: The epa controls should be within 15% of the given value

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

CADMIUM-GF IN LOVOL FILTERS

Operating Range = .00200 to 1.000 ug/filter

IN - RUN DUPLICATES

| | | | | | |
|-------|---------|------------------|------------------|-----------------|---------|
| Range | <.00200 | .00200 to 0.2000 | 0.2000 to 0.5000 | 0.5000 to 1.000 | > 1.000 |
| no. | 5 | 11 | 2 | 0 | 0 |
| s.w. | | 0.00400 | 0.02040 | 0.00000 | |
| mean | | 0.0390 | 0.2190 | 0.0000 | |

QA CONTROL SAMPLES

| SAMPLE I.D. | NO. | MEAN | STD. DEV. | R.S.D. |
|-------------|-----|---------|-----------|--------|
| epal | 64 | 0.04000 | 0.00500 | 12.50 |
| epah | 32 | 0.3500 | 0.03400 | 9.71 |

BLANKS

| BLANK I.D. | NO. | MEAN | STD. DEV. |
|------------|-----|--------|-----------|
| blk | 61 | .03000 | .04100 |

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Calcium TEST CODE: CAUR SAMPLE TYPE: Lovol filter
UNIT: Waters Unit SUPERVISOR: P. N. Vijan

METHOD CODE: 527AF2
REVISION NO: 1 DATE: Sept 1986
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required-one filter
Container-stored in a plastic petri dish
Preservative-
Other-

SAMPLE PREPARATION: Partial Extn.-yes Total Extn.- % Extracted-
Procedure-The exposed Lovol filter is extracted with 50 ml of double distilled water in an ultrasonic bath for 20 min. Twenty ml. of the water extract is taken for anion & cation analysis. The filter and the water extract are separated by decanting solution into separate tube. The water solution is preserved with .3ml of conc. HNO₃. The filter and insoluble contents are further extracted in 50 ml of .8N HNO₃ in a closed tube overnight at 90C. Both solutions are analyzed by ICP/ES. The results from the two solutions are combined in reporting in the units of ug/filter.

INTERFERENCES: Unknown

REPORTING RESULTS: ug per filter
INSTRUMENTATION: Jarrel Ash Atom-Comp-ICP Model 975

Calibration Range: 0 to 50 mg/L
Resolution: 0.02 mg/L
Sensitivity:
Instrument Detection Limit: .02 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-0 to 500 µg/filter (µg/Fi)

Accuracy-

Precision of Controls-

A

B

mean
std. dev.
R.S.D.

Precision of Duplicates-low range

mid range

high range

s.d. 1.03
mean 46.5

W 2 µg/Fi

T 10 µg/Fi

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

CALCIUM

IN LOVOL FILTERS

Operating Range = 2.0 to 500.0

IN - RUN DUPLICATES

| | | | | | | |
|-------|------|-------|----------|----------------|----------------|--------|
| Range | <2.0 | 2.0 | to 100.0 | 100.0 to 250.0 | 250.0 to 500.0 | >500.0 |
| no. | 2 | 6 | 0 | 0 | 0 | |
| s.w. | | 1.03 | 0.00 | 0.00 | | |
| mean | | 46.46 | 0.00 | 0.00 | | |

QA CONTROL SAMPLES

| | | | | |
|-------------|-----|------|-----------|--------|
| SAMPLE I.D. | NO. | MEAN | STD. DEV. | R.S.D. |
|-------------|-----|------|-----------|--------|

BLANKS

| | | | |
|------------|-----|------|-----------|
| BLANK I.D. | NO. | MEAN | STD. DEV. |
| rb | 18 | 0.00 | 0.00 |

DATE 87/11/03

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Copper
UNIT: Waters Unit

TEST CODE: CUUR SAMPLE TYPE: Lovol filter
SUPERVISOR: P. N. Vijan

METHOD CODE: 527AF2

REVISION NO: 1

DATE: Sept 1986

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-one filter
Container-stored in a plastic petri dish
Preservative-
Other-

SAMPLE PREPARATION: Partial Extn.-yes Total Extn.- % Extracted-

Procedure-The exposed lovol filter is extracted with 50 ml of double distilled water in an ultrasonic bath for 20 min. Twenty ml. of the water extract is taken for anion & cation analysis. The filter and the water extract are separated by decanting solution into separate tube. The water solution is preserved with 1% ultrapure HNO₃. The filter and insoluble contents are further extracted in 50 ml of .16N HNO₃ in a closed tube overnight at 90°C. Both solutions are analyzed by GFAAS. The results from the two solutions are combined in reporting in the units of ug/filter.

90 µL of sample is injected into the furnace and 50 lpm is the inter. argon flow at atomization.

INTERFERENCES: Unknown

REPORTING RESULTS: ug per filter

INSTRUMENTATION: Perkin Elmer 603 or 2380 Atomic Absorption Spectrophotometer with a HGA400 or HGA500 furnace and AS40 autosampler

Calibration Range: 0 to .060 mg/L

Resolution: 0.001 Abs. Units

Sensitivity: .030 mg/L = .250 Abs Units- Peak Height Mode

Instrument Detection Limit: .0002 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-0 to 6 µg/filter (µg/Fi)

Accuracy-

Precision of Controls-

| | A | B |
|-----------|-------|-------|
| mean | .360 | 3.43 |
| std. dev. | .035 | .186 |
| R.S.D. | 9.72% | 5.42% |

Precision of Duplicates-low range mid range high range

s.d. .022
mean .35

W .02 µg/Fi

T .10 µg/Fi

CONTROL LIMITS: The epa controls should be within 15% of the given value

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

COPPER-GF IN LOVOL FILTERS

Operating Range = .02000to 6.000 ug/filter

IN - RUN DUPLICATES

| | | | | | |
|-------|---------|----------------|----------------|----------------|---------|
| Range | <.02000 | .02000to1.2000 | 1.2000to3.0000 | 3.0000to 6.000 | > 6.000 |
| no. | 1 | 11 | 0 | 0 | 0 |
| s.w. | | 0.02200 | 0.00000 | 0.00000 | |
| mean | | 0.3500 | 0.0000 | 0.0000 | |

QA CONTROL SAMPLES

| SAMPLE I.D. | NO. | MEAN | STD. DEV. | R.S.D. |
|-------------|-----|---------|-----------|--------|
| epal | 75 | 0.36000 | 0.03500 | 9.72 |
| epah | 31 | 3.4300 | 0.18600 | 5.42 |

BLANKS

| BLANK I.D. | NO. | MEAN | STD. DEV. |
|------------|-----|--------|-----------|
| blk | 91 | .12000 | .16300 |

DATE 87/10/07

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Iron
UNIT:Waters Unit

TEST CODE:FEUR SAMPLE TYPE:Lovol filter
SUPERVISOR:P. N. Vijan

METHOD CODE:527AF2

REVISION NO:1

DATE:Sept 1986

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-one filter
Container-stored in a plastic petri dish
Preservative-
Other-

SAMPLE PREPARATION:Partial Extn.-yes Total Extn.- % Extracted-

Procedure-The exposed lovol filter is extracted with 50 ml of double distilled water in an ultrasonic bath for 20 min. Twenty ml. of the water extract is taken for anion & cation analysis. The filter and the water extract are separated by decanting solution into separate tube. The water solution is preserved with 1% ultrapure HNO₃. The filter and insoluble contents are further extracted in 50 ml of .16N HNO₃ in a closed tube overnight at 90°C. Both solutions are analyzed by GFAAS. The results from the two solutions are combined in reporting in the units of ug/filter.

20 µL of sample is injected into the furnace and 50 lpm is the inter. argon flow at atomization.

INTERFERENCES:Unknown

REPORTING RESULTS:ug per filter

INSTRUMENTATION:Perkin Elmer 603 or 2380 Atomic Absorption Spectrophotometer with a HGA400 or HGA500 furnace and AS40 autosampler

Calibration Range:0 to .10 mg/L

Resolution:0.001 Abs. Units

Sensitivity:.03 mg/L = .200 Abs Units- Peak Height Mode

Instrument Detection Limit: .001 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-0 to 10 µg/filter(µg/Fi)

Accuracy-

Precision of Controls-

| | A | B |
|-----------|-------|-------|
| mean | .780 | 7.95 |
| std. dev. | .061 | .507 |
| R.S.D. | 7.82% | 6.38% |

Precision of Duplicates-low range mid range high range

s.d. .050

mean .80

W .1 µg/Fi

T .5 µg/Fi

CONTROL LIMITS:The epa controls should be within 15% of the given value

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

IRON-GF

IN LOVOL FILTERS

Operating Range = .10000to 10.000 ug/filter

IN - RUN DUPLICATES

| | | | | | |
|-------|---------|----------------|----------------|----------------|---------|
| Range | <.10000 | .10000to2.0000 | 2.0000to5.0000 | 5.0000to10.000 | >10.000 |
| no. | 3 | 9 | 0 | 0 | 2 |
| s.w. | | 0.05000 | 0.00000 | 0.00000 | |
| mean | | 0.8000 | 0.0000 | 0.0000 | |

QA CONTROL SAMPLES

| SAMPLE I.D. | NO. | MEAN | STD. DEV. | R.S.D. |
|-------------|-----|---------|-----------|--------|
| epal | 49 | 0.78000 | 0.06400 | 8.21 |
| epah | 43 | 7.9500 | 0.50900 | 6.40 |

BLANKS

| BLANK I.D. | NO. | MEAN | STD. DEV. |
|------------|-----|--------|-----------|
| blk | 49 | .27000 | .21100 |

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Lead
UNIT:Waters Unit

TEST CODE:PBUR SAMPLE TYPE:Lovol filter
SUPERVISOR:P. N. Vijan

METHOD CODE:527AF2

REVISION NO:1

DATE:Sept 1986

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-one filter
Container-stored in a plastic petri dish
Preservative-
Other-

SAMPLE PREPARATION:Partial Extn.-yes Total Extn.- % Extracted-

Procedure-The exposed lovol filter is extracted with 50 ml of double distilled water in an ultrasonic bath for 20 min. Twenty ml. of the water extract is taken for anion &cation analysis. The filter and the water extract are separated by decanting solution into separate tube. The water solution is preserved with 1% ultrapure HNO₃. The filter and insoluble contents are further extracted in 50 ml of .16N HNO₃ in a closed tube overnight at 90°C. Both solutions are analyzed by GFAAS. The results from the two solutions are combined in reporting in the units of ug/filter.

20 µL of sample is injected into the furnace and 50 lpm is the inter. argon flow at atomization.

INTERFERENCES:Unknown

REPORTING RESULTS:ug per filter

INSTRUMENTATION:Perkin Elmer 603 or 2380 Atomic Absorption Spectrophotometer with a HGA400 or HGA500 furnace and AS40 autosampler

Calibration Range:0 to .10 mg/L

Resolution:0.001 Abs. Units

Sensitivity: 0.03 mg/L = .200 Abs Units- Peak Height Mode

Instrument Detection Limit: .001 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-0 to 10 µg/filter(µg/Fi)

Accuracy-

Precision of Controls-

| | A | B |
|-----------|-------|-------|
| mean | .435 | 4.345 |
| std. dev. | .046 | .571 |
| R.S.D. | 10.7% | 13.1% |

Precision of Duplicates-low range mid range high range

| | | |
|------|------|------|
| s.d. | .045 | .220 |
| mean | .550 | 4.60 |

4.60

W .1 µg/Fi

T .5 µg/Fi

CONTROL LIMITS:The epa controls should be within 15% of the given value

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

LEAD-GF

IN LOVOL FILTERS

Operating Range = .10000to 10.000 ug/filter

IN - RUN DUPLICATES

| | | | | | |
|-------|---------|----------------|----------------|----------------|---------|
| Range | <.10000 | .10000to2.0000 | 2.0000to5.0000 | 5.0000to10.000 | >10.000 |
| no. | 5 | 9 | 2 | 0 | 1 |
| s.w. | | 0.04000 | 0.22000 | 0.00000 | |
| mean | | 0.5500 | 4.6000 | 0.0000 | |

QA CONTROL SAMPLES

| SAMPLE I.D. | NO. | MEAN | STD. DEV. | R.S.D. |
|-------------|-----|---------|-----------|--------|
| epal | 73 | 0.44000 | 0.04600 | 10.45 |
| epah | 63 | 4.3600 | 0.60800 | 13.94 |

BLANKS

| BLANK I.D. | NO. | MEAN | STD. DEV. |
|------------|-----|--------|-----------|
| blk | 71 | .26000 | .25500 |

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Magnesium TEST CODE: MGUR SAMPLE TYPE: Lovol filter
UNIT: Waters Unit SUPERVISOR: P. N. Vijan

METHOD CODE: 527AF2

REVISION NO: 1

DATE: Sept 1986

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-one filter
Container-stored in a plastic petri dish
Preservative-
Other-

SAMPLE PREPARATION: Partial Extn.-yes Total Extn.- % Extracted-

Procedure-The exposed lovol filter is extracted with 50 ml of double distilled water in an ultrasonic bath for 20 min. Twenty ml. of the water extract is taken for anion & cation analysis. The filter and the water extract are separated by decanting solution into separate tube. The water solution is preserved with .3ml of conc. HNO₃. The filter and insoluble contents are further extracted in 50 ml of .8N HNO₃ in a closed tube overnight at 90C. Both solutions are analyzed by ICP/ES. The results from the two solutions are combined in reporting in the units of ug/filter.

INTERFERENCES: Unknown

REPORTING RESULTS: ug per filter

INSTRUMENTATION: Jarrel Ash Atom-Comp-ICP Model 975

Calibration Range: 0 to 50 mg/L

Resolution: 0.02 mg/L

Sensitivity:

Instrument Detection Limit: .02 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-0 to 500 µg/filter (µg/Fi)

Accuracy-

Precision of Controls-

A

B

mean

std. dev.

R.S.D.

Precision of Duplicates-low range

mid range

high range

s.d.

.19

mean

9.1

W 2 µg/Fi

T 10 µg/Fi

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

MAGNESIUM IN LOVOL FILTERS

Operating Range = 2.0 to 500.0 ug/filter

IN - RUN DUPLICATES

| | | | | | | |
|-------|------|------|---------|---------------|---------------|--------|
| Range | <2.0 | 2.0 | to100.0 | 100.0 to250.0 | 250.0 to500.0 | >500.0 |
| no. | 2 | 5 | 0 | 0 | 0 | |
| s.w. | | 0.19 | 0.00 | 0.00 | | |
| mean | | 9.05 | 0.00 | 0.00 | | |

QA CONTROL SAMPLES

| | | | | |
|-------------|-----|------|-----------|--------|
| SAMPLE I.D. | NO. | MEAN | STD. DEV. | R.S.D. |
|-------------|-----|------|-----------|--------|

BLANKS

| | | | |
|------------|-----|------|-----------|
| BLANK I.D. | NO. | MEAN | STD. DEV. |
| rb | 18 | 0.00 | 0.00 |

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Manganese TEST CODE: MNUR SAMPLE TYPE: Lovol filter
UNIT: Waters Unit SUPERVISOR: P. N. Vijan

METHOD CODE: 527AF2

REVISION NO: 1

DATE: Sept 1986

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required - one filter
Container - stored in a plastic petri dish
Preservative -
Other -

SAMPLE PREPARATION: Partial Extn. - yes Total Extn. - % Extracted -

Procedure - The exposed Lovol filter is extracted with 50 ml of double distilled water in an ultrasonic bath for 20 min. Twenty ml. of the water extract is taken for anion & cation analysis. The filter and the water extract are separated by decanting solution into separate tube. The water solution is preserved with 1% ultrapure HNO₃. The filter and insoluble contents are further extracted in 50 ml of .16N HNO₃ in a closed tube overnight at 90°C. Both solutions are analyzed by GFAAS. The results from the two solutions are combined in reporting in the units of ug/filter.

20 µL of sample is injected into the furnace and 50 lpm is the inter. argon flow at atomization.

INTERFERENCES: Unknown

REPORTING RESULTS: ug per filter

INSTRUMENTATION: Perkin Elmer 603 or 2380 Atomic Absorption Spectrophotometer with a HGA400 or HGA500 furnace and AS40 autosampler

Calibration Range: 0 to .06 mg/L

Resolution: 0.001 Abs. Units

Sensitivity: .010 mg/L = .210 Abs Units - Peak Height Mode

Instrument Detection Limit: .001 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range - 0 to 6 µg/filter (µg/Fi)

Accuracy -

Precision of Controls -

| | A | B |
|-----------|-------|-------|
| mean | .340 | 3.57 |
| std. dev. | .0415 | .471 |
| R.S.D. | 12.2% | 13.2% |

Precision of Duplicates - low range mid range high range

s.d. .010

mean .40

W .1 µg/Fi

T .5 µg/Fi

CONTROL LIMITS: The epa controls should be within 15% of the given value

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

MANGANESE-GF IN LOVOL FILTERS

Operating Range = .10000to 6.000 ug/filter

IN - RUN DUPLICATES

| | | | | | |
|-------|---------|----------------|----------------|----------------|---------|
| Range | <.10000 | .10000to1.2000 | 1.2000to3.0000 | 3.0000to 6.000 | > 6.000 |
| no. | 4 | 11 | 2 | 1 | 0 |
| s.w. | | 0.01000 | 0.00000 | 0.00000 | |
| mean | | 0.4000 | 1.7000 | 4.5000 | |

QA CONTROL SAMPLES

| SAMPLE I.D. | NO. | MEAN | STD. DEV. | R.S.D. |
|-------------|-----|---------|-----------|--------|
| epal | 67 | 0.34000 | 0.04200 | 12.35 |
| epah | 55 | 3.5800 | 0.47400 | 13.24 |

BLANKS

| BLANK I.D. | NO. | MEAN | STD. DEV. |
|------------|-----|--------|-----------|
| blk | 21 | .44000 | .36900 |

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Nickel TEST CODE: NIUR SAMPLE TYPE: Lovol filter
UNIT: Waters Unit SUPERVISOR: P. N. Vijan

METHOD CODE: 527AF2

REVISION NO: 1

DATE: Sept 1986

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-one filter
Container-stored in a plastic petri dish
Preservative-
Other-

SAMPLE PREPARATION: Partial Extn.-yes Total Extn.- % Extracted-

Procedure-The exposed lovol filter is extracted with 50 ml of double distilled water in an ultrasonic bath for 20 min. Twenty ml. of the water extract is taken for anion & cation analysis. The filter and the water extract are separated by decanting solution into separate tube. The water solution is preserved with 1% ultrapure HNO₃. The filter and insoluble contents are further extracted in 50 ml of .16N HNO₃ in a closed tube overnight at 90°C. Both solutions are analyzed by GFAAS. The results from the two solutions are combined in reporting in the units of ug/filter.

90 µL of sample is injected into the furnace and 0 lpm is the internal argon flow at atomization.

INTERFERENCES: Unknown

REPORTING RESULTS: ug per filter

INSTRUMENTATION: Perkin Elmer 603 or 2380 Atomic Absorption Spectrophotometer with a HGA400 or HGA500 furnace and AS40 autosampler

Calibration Range: 0 to .030 mg/L

Resolution: 0.001 Abs. Units

Sensitivity: .010 mg/L = .150 Abs Units- Peak Height Mode

Instrument Detection Limit: .0002 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-0 to 3 µg/filter (µg/Fi)

Accuracy-

Precision of Controls-

| | A | B |
|-----------|-------|-------|
| mean | .220 | 2.10 |
| std. dev. | .0150 | .175 |
| R.S.D. | 6.82% | 8.33% |

Precision of Duplicates-low range mid range high range

| | | |
|------|------|------|
| s.d. | .011 | .028 |
| mean | .115 | .980 |

W .02 µg/Fi

T .10 µg/Fi

CONTROL LIMITS: The epa controls should be within 15% of the given value

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

NICKEL-GF IN LOVOL FILTERS

Operating Range = .02000 to 3.000 ug/filter

IN - RUN DUPLICATES

Range <.02000 .02000 to 0.6000 0.6000 to 1.5000 1.5000 to 3.000 > 3.000

| | | | | | |
|------|---|---------|---------|---------|---|
| no. | 4 | 12 | 1 | 0 | 0 |
| s.w. | | 0.01050 | 0.02800 | 0.00000 | |
| mean | | 0.1150 | 0.9800 | 0.0000 | |

QA CONTROL SAMPLES

| SAMPLE I.D. | NO. | MEAN | STD. DEV. | R.S.D. |
|-------------|-----|---------|-----------|--------|
| epal | 62 | 0.22000 | 0.01500 | 6.82 |
| epah | 43 | 2.1000 | 0.17500 | 8.33 |

BLANKS

| BLANK I.D. | NO. | MEAN | STD. DEV. |
|------------|-----|--------|-----------|
| blk | 83 | .14000 | .23300 |

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Vanadium TEST CODE: VVUR SAMPLE TYPE: Lovol filter
UNIT: Waters Unit SUPERVISOR: P. N. Vijan

METHOD CODE: 527AF2

REVISION NO: 1

DATE: Sept 1986

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-one filter
Container-stored in a plastic petri dish
Preservative-
Other-

SAMPLE PREPARATION: Partial Extn.-yes Total Extn.- % Extracted-

Procedure-The exposed lovol filter is extracted with 50 ml of double distilled water in an ultrasonic bath for 20 min. Twenty ml. of the water extract is taken for anion & cation analysis. The filter and the water extract are separated by decanting solution into separate tube. The water solution is preserved with 1% ultrapure HNO₃. The filter and insoluble contents are further extracted in 50 ml of .16N HNO₃ in a closed tube overnight at 90°C. Both solutions are analyzed by GFAAS. The results from the two solutions are combined in reporting in the units of ug/filter.

90 µL of sample is injected into the furnace and 0 lpm is the internal argon flow at atomization.

INTERFERENCES: Unknown

REPORTING RESULTS: ug per filter

INSTRUMENTATION: Perkin Elmer 603 or 2380 Atomic Absorption Spectrophotometer with a HGA400 or HGA500 furnace and AS40 autosampler

Calibration Range: 0 to .01 mg/L

Resolution: 0.001 Abs. Units

Sensitivity: .002 mg/L = .018 Abs Units- Peak Height Mode

Instrument Detection Limit: .0004 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-0 to 1 µg/filter (µg/Fi)

Accuracy-

Precision of Controls-

| | A | B |
|-----------|-------|-------|
| mean | .930 | 8.63 |
| std. dev. | .079 | .56 |
| R.S.D. | 8.44% | 6.49% |

Precision of Duplicates-low range mid range high range

s.d. .010 .020

mean .10 .30

W .05 µg/Fi

T .25 µg/Fi

CONTROL LIMITS: The epa controls should be within 15% of the given value

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

VANADIUM-GF IN LOVOL FILTERS

Operating Range = .04000to 1.000 ug/filter

IN - RUN DUPLICATES

Range <.04000 .04000to0.2000 0.2000to0.5000 0.5000to 1.000 > 1.000

| | | | | | |
|------|----|---------|---------|---------|---|
| no. | 12 | 5 | 1 | 0 | 0 |
| s.w. | | 0.01000 | 0.02000 | 0.00000 | |
| mean | | 0.1000 | 0.3000 | 0.0000 | |

QA CONTROL SAMPLES

| SAMPLE I.D. | NO. | MEAN | STD. DEV. | R.S.D. |
|-------------|-----|---------|-----------|--------|
| epal | 48 | 0.93000 | 0.07800 | 8.39 |
| epah | 4 | 8.6300 | 0.56000 | 6.49 |

BLANKS

| BLANK I.D. | NO. | MEAN | STD. DEV. |
|------------|-----|--------|-----------|
| blk | 16 | .12000 | .08900 |

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Zinc TEST CODE: ZNUR SAMPLE TYPE: Lovol filter
UNIT: Waters Unit SUPERVISOR: P. N. Vijan

METHOD CODE: 527AF2

REVISION NO: 1

DATE: Sept 1986

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required-one filter
Container-stored in a plastic petri dish
Preservative-
Other-

SAMPLE PREPARATION: Partial Extn.-yes Total Extn.- % Extracted-

Procedure-The exposed lovol filter is extracted with 50 ml of double distilled water in an ultrasonic bath for 20 min. Twenty ml. of the water extract is taken for anion & cation analysis. The filter and the water extract are separated by decanting solution into separate tube. The water solution is preserved with 1% ultrapure HNO₃. The filter and insoluble contents are further extracted in 50 ml of .16N HNO₃ in a closed tube overnight at 90°C. Both solutions are analyzed by GFAAS. The results from the two solutions are combined in reporting in the units of ug/filter.

20 µL of sample is injected into the furnace and 50 lpm is the inter. argon flow at atomization.

INTERFERENCES: Unknown

REPORTING RESULTS: ug per filter

INSTRUMENTATION: Perkin Elmer 603 or 2380 Atomic Absorption Spectrophotometer with a HGA400 or HGA500 furnace and AS40 autosampler

Calibration Range: 0 to .100 mg/L

Resolution: 0.001 Abs. Units

Sensitivity: .030 mg/L = .600 Abs Units- Peak Height Mode

Instrument Detection Limit: .001 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range-0 to 10 µg/filter (µg/Fi)

Accuracy-

Precision of Controls-

| | A | B |
|-----------|-------|-------|
| mean | .410 | 3.85 |
| std. dev. | .049 | .354 |
| R.S.D. | 12.0% | 9.19% |

Precision of Duplicates-low range mid range high range

| | | |
|------|------|------|
| s.d. | .040 | .140 |
| mean | .80 | 3.60 |

W .1 µg/Fi

T .5 µg/Fi

CONTROL LIMITS: The epa controls should be within 15% of the given value

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

ZINC-GF

IN LOVOL FILTERS

Operating Range = .10000 to 10.000 ug/filter

IN - RUN DUPLICATES

Range <.10000 .10000 to 2.0000 2.0000 to 5.0000 5.0000 to 10.000 >10.000

| | | | | | |
|------|---|---------|---------|---------|---|
| no. | 9 | 6 | 1 | 0 | 1 |
| s.w. | | 0.04000 | 0.14000 | 0.00000 | |
| mean | | 0.8000 | 3.6000 | 0.0000 | |

QA CONTROL SAMPLES

| SAMPLE I.D. | NO. | MEAN | STD. DEV. | R.S.D. |
|-------------|-----|---------|-----------|--------|
| epal | 65 | 0.41000 | 0.04900 | 11.95 |
| epah | 30 | 3.8500 | 0.35400 | 9.19 |

BLANKS

| BLANK I.D. | NO. | MEAN | STD. DEV. |
|------------|-----|--------|-----------|
| blk | 44 | .18000 | .12100 |

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Aluminum TEST CODE: ALPDT SAMPLE TYPE: Precipdep tot
UNIT: Water SUPERVISOR: P. Vijan

METHOD CODE: 520AF2
REVISION NO: Original DATE: 1982
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 100 ml
Container- Polyethylene bag (approx. 10x20 cm) with plastic screw top
Preservative- Conc HNO₃ (0.25%) - ultra-pure
Other- Sample kept refrigerated until preserved

SAMPLE PREPARATION: Partial Extn.- Total Extn.-Yes % Extracted-
Procedure- Transfer 50 ml sample from sample bag to calibrated
polyethhylene tube and preserve with 1% nitric acid.
Reduce to less than 5 ml by evaporation in an oven and dilute to
exactly the 5 ml mark at room temperature.
Two filtered composites, one spiked at a higher level, are also
taken through this preconcentration procedure, as controls.
A typical run consists of 40 test tubes including blanks, controls,
digested standards and samples.
Al in the solutions is determined as part of a multielement
measurement system.

INTERFERENCES: None

REPORTING RESULTS: To 3 decimals

INSTRUMENTATION: Jarell Ash Atom-Comp -ICP/ES Model 975, 0.75 m,
with autosampler and DEC computer for simultaneous concentration
printout. PET interface for data message, storage & transfer to computer

Calibration Range: 0 to 500 µg/ml

Resolution: 0.01 µg/ml

Sensitivity:

Instrument Detection Limit: 0.08 µg/ml

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0 to 20 µg/ml

Accuracy-93% at 0.68 µg/ml

Precision of Controls-

A

B

mean

std. dev.

R.S.D.

Precision of Duplicates-low range

mid range

high range

s.d. 0.0012

0.0062

0.0083

mean

W

T

CONTROL LIMITS:

REMARKS: Occasional use of graphite furnace AAS is permitted as
backup instrument in event of equipment failure, specific problem
solving and to expedite analyses if fewer than four metals are to be
determined.

SUMMARY REPORT OF QUALITY CONTROL DATA

ALUMINIUM IN PRECIPITATION

Operating Range = .00200 to 0.500 mg/L

IN - RUN DUPLICATES

| Range | <.00200 | .00200 to 0.1000 | 0.1000 to 0.2500 | 0.2500 to 0.500 | > 0.500 |
|-------|---------|------------------|------------------|-----------------|---------|
| no. | 0 | 45 | 2 | 0 | 0 |
| s.w. | | 0.00540 | 0.07000 | 0.00000 | |
| mean | | 0.0294 | 0.1678 | 0.0000 | |

QA CONTROL SAMPLES

| SAMPLE I.D. | NO. | MEAN | STD. DEV. | R.S.D. |
|-------------|-----|---------|-----------|--------|
| std | 11 | 0.00660 | 0.00638 | 96.67 |
| qcstd | 16 | 0.2505 | 0.03846 | 15.35 |

BLANKS

| BLANK I.D. | NO. | MEAN | STD. DEV. |
|------------|-----|--------|-----------|
| BLK | 3 | .19280 | .10037 |

DATE 87/08/07

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Aluminum TEST CODE: ALPDT SAMPLE TYPE: Precipdep tot
UNIT: Water SUPERVISOR: P. Vijan

METHOD CODE: 520AF2
REVISION NO: Original DATE: 1982
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 100 ml
Container- Polyethylene bag (approx. 10x20 cm) with plastic screw top
Preservative- Conc HNO₃ (0.25%) - ultra-pure
Other- Sample kept refrigerated until preserved

SAMPLE PREPARATION: Partial Extn.- Total Extn.-Yes % Extracted-
Procedure- Transfer 50 ml sample from sample bag to calibrated
polyethylene test tube and preserve with 1% nitric acid.
Analyze by graphite furnace a.a.s. with 20 µL of sample, an
argon internal flow of 0 lpm, and in peak height mode. The other
furnace conditions are set according to manual specifications.

INTERFERENCES: None

REPORTING RESULTS: To 3 decimals

INSTRUMENTATION: Perkin-Elmer Models 2380 or 603 AAS with PE 400 or
PE 500 graphite furnace atomizer systems, and AS 40 autosamplers. Both
interfaced with PET computers for data handling.

Calibration Range: 0.0 to .100 mg/L

Resolution: 0.001 Abs. Units

Sensitivity: 0.030 mg/L = .200 Abs. Units

Instrument Detection Limit: 0.005 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.0 to 0.100 mg/L

Accuracy-

Precision of Controls-

| | A | B |
|-----------|-----------|-------|
| mean | .0069mg/L | .080 |
| std. dev. | .0014mg/L | .0068 |
| R.S.D. | 20.4 % | 8.5 % |

| Precision of Duplicates- | low range | mid range | high range |
|--------------------------|-----------|-----------|------------|
| s.d. | .0013 | .0020 | .0022 |
| mean | .014 | .031 | .077 |

W .001 mg/L T .010 mg/L

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

ALUMINUM-GF IN PRECIPITATION

Operating Range = .00100to 0.100 mg/L

IN - RUN DUPLICATES

| Range | <.00100 | .00100to0.0200 | 0.0200to0.0500 | 0.0500to 0.100 | > 0.100 |
|-------|---------|----------------|----------------|----------------|---------|
| no. | 11 | 19 | 14 | 2 | 0 |
| s.w. | | 0.00130 | 0.00200 | 0.00220 | |
| mean | | 0.0140 | 0.0310 | 0.0770 | |

QA CONTROL SAMPLES

| SAMPLE I.D. | NO. | MEAN | STD. DEV. | R.S.D. |
|-------------|-----|---------|-----------|--------|
| epal | 22 | 0.00690 | 0.00141 | 20.43 |
| epah | 24 | 0.0804 | 0.00681 | 8.47 |
| std 4 | 25 | 0.0098 | 0.00222 | 22.65 |
| std 5 | 23 | 0.0215 | 0.00433 | 20.14 |

BLANKS

| BLANK I.D. | NO. | MEAN | STD. DEV. |
|------------|-----|--------|-----------|
| blk | 17 | .00630 | .00113 |

DATE 87/02/25

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Cadmium
UNIT: Water

TEST CODE: CDPDT SAMPLE TYPE: Precipdep tot
SUPERVISOR: P. Vijan

METHOD CODE: 520AF2

REVISION NO: Original

DATE: 1982

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 100 ml

Container- Polyethylene bag (approx. 10x20 cm) with plastic screw top

Preservative- Conc HNO₃ (0.25%) - ultra-pure

Other- Sample kept refrigerated until preserved

SAMPLE PREPARATION: Partial Extn.- Total Extn.-Yes % Extracted-

Procedure- Transfer 50 ml sample from sample bag to calibrated polyethhylene tube and preserve with 1% nitric acid.

Reduce to less than 5 ml by evaporation in an oven and dilute to exactly the 5 ml mark at room temperature.

Two filtered composites, one spiked at a higher level, are also taken through this preconcentration procedure, as controls.

A typical run consists of 40 test tubes including blanks, controls, digested standards and samples.

Cd in the solutions is determined as part of a multielement measurement system.

INTERFERENCES: None

REPORTING RESULTS: To 4 decimals

INSTRUMENTATION: Jarell Ash Atom-Comp -ICP/ES Model 975, 0.75 m, with autosampler and DEC computer for simultaneous concentration printout. PET interface for data message, storage & transfer to computer

Calibration Range: 0 to 100 µg/ml

Resolution:

Sensitivity:

Instrument Detection Limit: 0.005 µg/ml

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0 to 0.5 µg/ml

Accuracy-99% at 0.38 µg/ml

Precision of Controls-

A

B

mean

std. dev.

R.S.D.

Precision of Duplicates-low range

mid range

high range

s.d. 0.00002

0.00020

mean

W

T

CONTROL LIMITS:

REMARKS: Occasional use of graphite furnace AAS is permitted as backup instrument in event of equipment failure, specific problem solving and to expedite analyses if fewer than four metals are to be determined.

SUMMARY REPORT OF QUALITY CONTROL DATA

CADMIUM IN PRECIPITATION

Operating Range = .00010 to 0.010 mg/L

IN - RUN DUPLICATES

| Range | <.00010 | .00010 to 0.0020 | 0.0020 to 0.0050 | 0.0050 to 0.010 | > 0.010 |
|-------|---------|------------------|------------------|-----------------|---------|
| no. | 41 | 5 | 1 | 0 | 0 |
| s.w. | | 0.00020 | 0.00030 | 0.00000 | |
| mean | | 0.0003 | 0.0039 | 0.0000 | |

QA CONTROL SAMPLES

| SAMPLE I.D. | NO. | MEAN | STD. DEV. | R.S.D. |
|-------------|-----|---------|-----------|--------|
| std | 28 | 0.00280 | 0.00149 | 53.21 |
| qcstd | 17 | 0.1994 | 0.01839 | 9.22 |

BLANKS

| BLANK I.D. | NO. | MEAN | STD. DEV. |
|------------|-----|--------|-----------|
| BLK | 2 | .03410 | .04080 |

DATE 87/08/07

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Cadmium TEST CODE: CDPDT SAMPLE TYPE: Precipdep tot
UNIT: Water SUPERVISOR: P. Vijan

METHOD CODE: 520AF2
REVISION NO: Original DATE: 1982
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 100 ml
Container- Polyethylene bag (approx. 10x20 cm) with plastic screw top
Preservative- Conc HNO₃ (0.25%) - ultra-pure
Other- Sample kept refrigerated until preserved

SAMPLE PREPARATION: Partial Extn.- Total Extn.- Yes % Extracted-
Procedure- Transfer 50 ml sample from sample bag to calibrated
polyethylene test tube and preserve with 1% nitric acid.
Analyze by graphite furnace a.a.s. with 90 µL of sample, an
argon internal flow of 50 lpm, and in peak height mode. The other
furnace conditions are set according to manual specifications.

INTERFERENCES: None

REPORTING RESULTS: To 5 decimals

INSTRUMENTATION: Perkin-Elmer Models 2380 or 603 AAS with PE 400 or
PE 500 graphite furnace atomizer systems, and AS 40 autosamplers. Both
interfaced with PET computers for data handling.

Calibration Range: 0.0 to .010 mg/L

Resolution: 0.001 Abs. Units

Sensitivity: 0.005 mg/L = .450 Abs. Units

Instrument Detection Limit: 0.00002 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.0 to 0.010 mg/L

Accuracy-

Precision of Controls-

| | A | B |
|-----------|-----------|--------|
| mean | .0004mg/L | .0036 |
| std. dev. | .00004mgL | .0004 |
| R.S.D. | 10.0 % | 11.7 % |

Precision of Duplicates-low range mid range high range

s.d. .00001 .0088

mean .0002 .049

W .02 µg/L

T .10 µg/L

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

CADMIUM-GF IN PRECIPITATION

Operating Range = .00002 to 0.010 mg/L

IN - RUN DUPLICATES

| Range | <.00002 | .00002 to 0.0020 | 0.0020 to 0.0050 | 0.0050 to 0.010 | > 0.010 |
|-------|---------|------------------|------------------|-----------------|---------|
| no. | 18 | 39 | 0 | 0 | 0 |
| s.w. | | 0.00001 | 0.00000 | 0.00000 | |
| mean | | 0.0002 | 0.0000 | 0.0000 | |

QA CONTROL SAMPLES

| SAMPLE I.D. | NO. | MEAN | STD. DEV. | R.S.D. |
|-------------|-----|---------|-----------|--------|
| epal | 28 | 0.00040 | 0.00004 | 10.00 |
| epah | 7 | 0.0036 | 0.00042 | 11.67 |
| std 4 | 12 | 0.0102 | 0.00107 | 10.49 |
| std 5 | 0 | 0.0000 | 0.00000 | 0.00 |

BLANKS

| BLANK I.D. | NO. | MEAN | STD. DEV. |
|------------|-----|--------|-----------|
| blk | 0 | .00000 | .00000 |

DATE 87/02/25

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Copper
UNIT: Water

TEST CODE: CUPDT SAMPLE TYPE: Precipdep tot
SUPERVISOR: P. Vijan

METHOD CODE: 520AF2

REVISION NO: Original

DATE: 1982

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 100 ml

Container- Polyethylene bag (approx. 10x20 cm) with plastic screw top

Preservative- Conc HNO₃ (0.25%) - ultra-pure

Other- Sample kept refrigerated until preserved

SAMPLE PREPARATION: Partial Extn.- Total Extn.-Yes % Extracted-

Procedure- Transfer 50 ml sample from sample bag to calibrated polyethhylene tube and preserve with 1% nitric acid.

Reduce to less than 5 ml by evaporation in an oven and dilute to exactly the 5 ml mark at room temperature.

Two filtered composites, one spiked at a higher level, are also taken through this preconcentration procedure, as controls.

A typical run consists of 40 test tubes including blanks, controls, digested standards and samples.

Cu in the solutions is determined as part of a multielement measurement system.

INTERFERENCES: None

REPORTING RESULTS: To 3 decimals

INSTRUMENTATION: Jarell Ash Atom-Comp -ICP/ES Model 975, 0.75 m, with autosampler and DEC computer for simultaneous concentration printout. PET interface for data message, storage & transfer to computer

Calibration Range: 0 to 100 µg/ml

Resolution:

Sensitivity:

Instrument Detection Limit: 0.006 µg/ml

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0 to 1 µg/ml

Accuracy-91% at 0.31 µg/ml

Precision of Controls-

mean
std. dev.
R.S.D.

A

B

Precision of Duplicates-low range

mid range

high range

s.d. 0.0003

0.0005

0.0004

mean

W

T

CONTROL LIMITS:

REMARKS: Occasional use of graphite furnace AAS is permitted as backup instrument in event of equipment failure, specific problem solving and to expedite analyses if fewer than four metals are to be determined.

SUMMARY REPORT OF QUALITY CONTROL DATA

COPPER

IN PRECIPITATION

Operating Range = .00030 to 0.050 mg/L

IN - RUN DUPLICATES

| | | | | | |
|-------|---------|------------------|------------------|-----------------|---------|
| Range | <.00030 | .00030 to 0.0100 | 0.0100 to 0.0250 | 0.0250 to 0.050 | > 0.050 |
| no. | 8 | 37 | 0 | 2 | 0 |
| s.w. | | 0.00120 | 0.00000 | 0.00350 | |
| mean | | 0.0011 | 0.0000 | 0.0320 | |

QA CONTROL SAMPLES

| SAMPLE I.D. | NO. | MEAN | STD. DEV. | R.S.D. |
|-------------|-----|---------|-----------|--------|
| std | 26 | 0.00310 | 0.00089 | 28.71 |
| qcstd | 17 | 0.2199 | 0.01771 | 8.05 |

BLANKS

| BLANK I.D. | NO. | MEAN | STD. DEV. |
|------------|-----|--------|-----------|
| BLK | 20 | .01350 | .01492 |

DATE 87/08/07

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Copper TEST CODE: CUPDT SAMPLE TYPE: Precipdep tot
UNIT: Water SUPERVISOR: P. Vijan

METHOD CODE: 520AF2
REVISION NO: Original DATE: 1982
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 100 ml
Container- Polyethylene bag (approx. 10x20 cm) with plastic screw top
Preservative- Conc HNO₃ (0.25%) - ultra-pure
Other- Sample kept refrigerated until preserved

SAMPLE PREPARATION: Partial Extn.- Total Extn.-Yes % Extracted-
Procedure- Transfer 50 ml sample from sample bag to calibrated
polyethylene test tube and preserve with 1% nitric acid.
Analyze by graphite furnace a.a.s. with 90 µL of sample, an
argon internal flow of 50 lpm, and in peak height mode. The other
furnace conditions are set according to manual specifications.

INTERFERENCES: None

REPORTING RESULTS: To 4 decimals

INSTRUMENTATION: Perkin-Elmer Models 2380 or 603 AAS with PE 400 or
PE 500 graphite furnace atomizer systems, and AS 40 autosamplers. Both
interfaced with PET computers for data handling.

Calibration Range: 0.0 to .060 mg/L

Resolution: 0.001 Abs. Units

Sensitivity: 0.030 mg/L gives .250 Abs. Units

Instrument Detection Limit: 0.0002

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.0 to 0.06 mg/L

Accuracy- 91% at 0.31 µg/ml

Precision of Controls-

| | A | B |
|-----------|-----------|-------|
| mean | .0034mg/L | .0332 |
| std. dev. | .0002mg/L | .0024 |
| R.S.D. | 5.9 % | 7.1 % |

Precision of Duplicates-low range mid range high range

| | | |
|------|--------|--------|
| s.d. | 0.0001 | 0.0006 |
|------|--------|--------|

| | | |
|------|--------|--------|
| mean | 0.0015 | 0.0397 |
|------|--------|--------|

W .0002mg/L T .0020mg/L

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

COPPER-GF IN PRECIPITATION

Operating Range = .00020 to 0.060 mg/L

IN - RUN DUPLICATES

| Range | <.00020 | .00020 to 0.0120 | 0.0120 to 0.0300 | 0.0300 to 0.060 | > 0.060 |
|-------|---------|------------------|------------------|-----------------|---------|
| no. | 23 | 22 | 0 | 1 | 0 |
| s.w. | | 0.00011 | 0.00000 | 0.00057 | |
| mean | | 0.0015 | 0.0000 | 0.0397 | |

QA CONTROL SAMPLES

| SAMPLE I.D. | NO. | MEAN | STD. DEV. | R.S.D. |
|-------------|-----|---------|-----------|--------|
| epal | 25 | 0.00340 | 0.00020 | 5.88 |
| epah | 11 | 0.0332 | 0.00236 | 7.11 |
| std 4 | 22 | 0.0103 | 0.00151 | 14.66 |
| std 5 | 15 | 0.0209 | 0.00218 | 10.43 |

BLANKS

| BLANK I.D. | NO. | MEAN | STD. DEV. |
|------------|-----|--------|-----------|
| blk | 10 | .00020 | .00004 |

DATE 87/02/25

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Iron
UNIT: Water

TEST CODE: FEPDT SAMPLE TYPE: Precipdep tot
SUPERVISOR: P. Vijan

METHOD CODE: 520AF2
REVISION NO: Original
NATURE OF LAST REVISION:

DATE: 198



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Laboratory Library
125 Resource Rd.
Etobicoke, Ontario M9P 3V6
Canada

SAMPLE HANDLING:

Quantity Required- 100 ml
Container- Polyethylene bag (approx. 10x20 cm) with plastic screw top
Preservative- Conc HNO₃ (0.25%) - ultra-pure
Other- Sample kept refrigerated until preserved

SAMPLE PREPARATION: Partial Extn.- Total Extn.-Yes % Extracted-
Procedure- Transfer 50 ml sample from sample bag to calibrated
polyethhylene tube and preserve with 1% nitric acid.
Reduce to less than 5 ml by evaporation in an oven and dilute to
exactly the 5 ml mark at room temperature.
Two filtered composites, one spiked at a higher level, are also
taken through this preconcentration procedure, as controls.
A typical run consists of 40 test tubes including blanks, controls,
digested standards and samples.
Fe in the solutions is determined as part of a multielement
measurement system.

INTERFERENCES: None

REPORTING RESULTS: To 3 decimals

INSTRUMENTATION: Jarell Ash Atom-Comp -ICP/ES Model 975, 0.75 m,
with autosampler and DEC computer for simultaneous concentration
printout. PET interface for data message, storage & transfer to computer

Calibration Range: 0 to 500 µg/ml

Resolution:

Sensitivity:

Instrument Detection Limit: 0.02 µg/ml

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0 to 20 µg/ml

Accuracy-110% at 0.88 µg/ml

Precision of Controls-

mean
std. dev.
R.S.D.

A

B

Precision of Duplicates-low range

s.d. 0.0018
mean

mid range
0.0061

high range
0.0129

W

T

CONTROL LIMITS:

REMARKS: Occasional use of graphite furnace AAS is permitted as
backup instrument in event of equipment failure, specific problem
solving and to expedite analyses if fewer than four metals are to be
determined.

SUMMARY REPORT OF QUALITY CONTROL DATA

IRON

IN PRECIPITATION

Operating Range = .00100to 0.500 mg/L

IN - RUN DUPLICATES

| Range | <.00100 | .00100to0.1000 | 0.1000to0.2500 | 0.2500to 0.500 | > 0.500 |
|-------|---------|----------------|----------------|----------------|---------|
| no. | 0 | 45 | 1 | 1 | 0 |
| s.w. | | 0.00610 | 0.00500 | 0.15950 | |
| mean | | 0.0216 | 0.1277 | 0.3799 | |

QA CONTROL SAMPLES

| SAMPLE I.D. | NO. | MEAN | STD. DEV. | R.S.D. |
|-------------|-----|---------|-----------|--------|
| std | 24 | 0.01850 | 0.07496 | 405.19 |
| qcstd | 17 | 0.1940 | 0.04500 | 23.20 |

BLANKS

| BLANK I.D. | NO. | MEAN | STD. DEV. |
|------------|-----|--------|-----------|
| BLK | 9 | .09190 | .07532 |

DATE 87/08/07

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Iron
UNIT: Water

TEST CODE: FEPDT SAMPLE TYPE: Precipdep tot
SUPERVISOR: P. Vijan

METHOD CODE: 520AF2
REVISION NO: Original
NATURE OF LAST REVISION:

DATE: 1982

SAMPLE HANDLING:

Quantity Required- 100 ml
Container- Polyethylene bag (approx. 10x20 cm) with plastic screw top
Preservative- Conc HNO₃ (0.25%) - ultra-pure
Other- Sample kept refrigerated until preserved

SAMPLE PREPARATION: Partial Extn.- Total Extn.- Yes % Extracted-
Procedure- Transfer 50 ml sample from sample bag to calibrated polyethylene test tube and preserve with 1% nitric acid.
Analyze by graphite furnace a.a.s. with 20 µL of sample, an argon internal flow of 50 lpm, and in peak height mode. The other furnace conditions are set according to manual specifications.

INTERFERENCES: None

REPORTING RESULTS: To 3 decimals

INSTRUMENTATION: Perkin-Elmer Models 2380 or 603 AAS with PE 400 or PE 500 graphite furnace atomizer systems, and AS 40 autosamplers. Both interfaced with PET computers for data handling.

Calibration Range: 0.0 to .100 mg/L

Resolution: 0.001 Abs. Units

Sensitivity: 0.030 mg/L gives .200 Abs. Units

Instrument Detection Limit: 0.001 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.0 to 0.100 mg/L

Accuracy-

Precision of Controls-

| | A | B |
|-----------|-----------|-------|
| mean | .0076mg/L | .079 |
| std. dev. | .0007mg/L | .0043 |
| R.S.D. | 8.6 % | 5.5 % |

| Precision of Duplicates- | low range | mid range | high range |
|--------------------------|-----------|-----------|------------|
| s.d. | .0014 | .0026 | |
| mean | .011 | .025 | |

W .001 mg/L

T .005 mg/L

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

IRON-GF

IN PRECIPITATION

Operating Range = .00100to 0.100 mg/L

IN - RUN DUPLICATES

| Range | <.00100 | .00100to0.0200 | 0.0200to0.0500 | 0.0500to 0.100 | > 0.100 |
|-------|---------|----------------|----------------|----------------|---------|
| no. | 10 | 22 | 6 | 0 | 1 |
| s.w. | | 0.00140 | 0.00260 | 0.00000 | |
| mean | | 0.0110 | 0.0250 | 0.0000 | |

QA CONTROL SAMPLES

| SAMPLE I.D. | NO. | MEAN | STD. DEV. | R.S.D. |
|-------------|-----|---------|-----------|--------|
| epal | 24 | 0.00760 | 0.00065 | 8.55 |
| epah | 23 | 0.0791 | 0.00432 | 5.46 |
| std 4 | 23 | 0.0100 | 0.00097 | 9.70 |
| std 5 | 26 | 0.0231 | 0.00249 | 10.78 |

BLANKS

| BLANK I.D. | NO. | MEAN | STD. DEV. |
|------------|-----|--------|-----------|
| blk | 46 | .00180 | .00076 |

DATE 87/02/25

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Lead
UNIT: Water

TEST CODE: PBPDT SAMPLE TYPE: Precipdep tot
SUPERVISOR: P. Vijan

METHOD CODE: 520AF2

REVISION NO: Original

DATE: 1982

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 100 ml

Container- Polyethylene bag (approx. 10x20 cm) with plastic screw top

Preservative- Conc HNO₃ (0.25%) - ultra-pure

Other- Sample kept refrigerated until preserved

SAMPLE PREPARATION: Partial Extn.- Total Extn.-Yes % Extracted-

Procedure- Transfer 50 ml sample from sample bag to calibrated polyethylene test tube and preserve with 1% nitric acid.

Analyze by graphite furnace a.a.s. with 20 µL of sample, an argon internal flow of 50 lpm, and in peak height mode. The other furnace conditions are set according to manual specifications.

INTERFERENCES: None

REPORTING RESULTS: To 3 decimals

INSTRUMENTATION: Perkin-Elmer Models 2380 or 603 AAS with PE 400 or PE 500 graphite furnace atomizer systems, and AS 40 autosamplers. Both interfaced with PET computers for data handling.

Calibration Range: 0.0 to .100 mg/L

Resolution: 0.001 Abs. Units

Sensitivity: 0.030 mg/L = .200 Abs. Units

Instrument Detection Limit: 0.001 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.0 to 0.100 mg/L

Accuracy-

Precision of Controls-

| | A | B |
|-----------|-----------|-------|
| mean | .0043mg/L | .044 |
| std. dev. | .0005mg/L | .0028 |
| R.S.D. | 11.1 % | 6.4 % |

Precision of Duplicates-low range mid range high range

s.d. .0004 .0002

mean .003 .015

W .001 mg/L

T .005 mg/L

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

LEAD-GF IN PRECIPITATION

Operating Range = .00100to 0.100 mg/L

IN - RUN DUPLICATES

| Range | <.00100 | .00100to0.0200 | 0.0200to0.0500 | 0.0500to 0.100 | > 0.100 |
|-------|---------|----------------|----------------|----------------|---------|
| no. | 14 | 38 | 0 | 0 | 0 |
| s.w. | | 0.00040 | 0.00000 | 0.00000 | |
| mean | | 0.0030 | 0.0000 | 0.0000 | |

QA CONTROL SAMPLES

| SAMPLE I.D. | NO. | MEAN | STD. DEV. | R.S.D. |
|-------------|-----|---------|-----------|--------|
| epal | 27 | 0.00430 | 0.00048 | 11.16 |
| epah | 26 | 0.0444 | 0.00282 | 6.35 |
| std 4 | 22 | 0.0111 | 0.00347 | 31.26 |
| std 5 | 15 | 0.0230 | 0.00286 | 12.43 |

BLANKS

| BLANK I.D. | NO. | MEAN | STD. DEV. |
|------------|-----|--------|-----------|
| blk | 27 | .00260 | .00114 |

DATE 87/02/25

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Manganese TEST CODE: MNPDT SAMPLE TYPE: Precipdep tot
UNIT: Water SUPERVISOR: P. Vijan

METHOD CODE: 520AF2
REVISION NO: Original DATE: 1982
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 100 ml
Container- Polyethylene bag (approx. 10x20 cm) with plastic screw top
Preservative- Conc HNO₃ (0.25%) - ultra-pure
Other- Sample kept refrigerated until preserved

SAMPLE PREPARATION: Partial Extn.- Total Extn.-Yes % Extracted-
Procedure- Transfer 50 ml sample from sample bag to calibrated
polyethhylene tube and preserve with 1% nitric acid.
Reduce to less than 5 ml by evaporation in an oven and dilute to
exactly the 5 ml mark at room temperature.
Two filtered composites, one spiked at a higher level, are also
taken through this preconcentration procedure, as controls.
A typical run consists of 40 test tubes including blanks, controls,
digested standards and samples.
Mn in the solutions is determined as part of a multielement
measurement system.

INTERFERENCES: None

REPORTING RESULTS: To 3 decimals

INSTRUMENTATION: Jarell Ash Atom-Comp -ICP/ES Model 975, 0.75 m,
with autosampler and DEC computer for simultaneous concentration
printout. PET interface for data message, storage & transfer to computer

Calibration Range: 0 to 100 µg/ml

Resolution:

Sensitivity:

Instrument Detection Limit: 0.003 µg/ml

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0 to 5 µg/ml

Accuracy-107% at 0.37 µg/ml

Precision of Controls-

A

B

mean

std. dev.

R.S.D.

Precision of Duplicates-low range

mid range

high range

s.d. 0.00011

0.00037

0.00040

mean

W

T

CONTROL LIMITS:

REMARKS: Occasional use of graphite furnace AAS is permitted as
backup instrument in event of equipment failure, specific problem
solving and to expedite analyses if fewer than four metals are to be
determined.

SUMMARY REPORT OF QUALITY CONTROL DATA

MANGANESE IN PRECIPITATION

Operating Range = .00020 to 0.030 mg/L

IN - RUN DUPLICATES

| | | | | | |
|-------|---------|------------------|------------------|-----------------|---------|
| Range | <.00020 | .00020 to 0.0060 | 0.0060 to 0.0150 | 0.0150 to 0.030 | > 0.030 |
| no. | 0 | 45 | 1 | 0 | 1 |
| s.w. | | 0.00060 | 0.00040 | 0.00000 | |
| mean | | 0.0029 | 0.0129 | 0.0000 | |

QA CONTROL SAMPLES

| SAMPLE I.D. | NO. | MEAN | STD. DEV. | R.S.D. |
|-------------|-----|---------|-----------|--------|
| std | 28 | 0.00300 | 0.00157 | 52.33 |
| qcstd | 17 | 0.2178 | 0.02089 | 9.59 |

BLANKS

| BLANK I.D. | NO. | MEAN | STD. DEV. |
|------------|-----|--------|-----------|
| BLK | 2 | .03740 | .04681 |

DATE 87/08/07

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Manganese TEST CODE: MNPDT SAMPLE TYPE: Precipdep tot
UNIT: Water SUPERVISOR: P. Vijan

METHOD CODE: 520AF2
REVISION NO: Original DATE: 1982
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 100 ml
Container- Polyethylene bag (approx. 10x20 cm) with plastic screw top
Preservative- Conc HNO₃ (0.25%) - ultra-pure
Other- Sample kept refrigerated until preserved

SAMPLE PREPARATION: Partial Extn.- Total Extn.-Yes % Extracted-
Procedure- Transfer 50 ml sample from sample bag to calibrated
polyethylene test tube and preserve with 1% nitric acid.
Analyze by graphite furnace a.a.s. with 20 µL of sample, an
argon internal flow of 50 lpm, and in peak height mode. The other
furnace conditions are set according to manual specifications.

INTERFERENCES: None

REPORTING RESULTS: To 3 decimals

INSTRUMENTATION: Perkin-Elmer Models 2380 or 603 AAS with PE 400 or
PE 500 graphite furnace atomizer systems, and AS 40 autosamplers. Both
interfaced with PET computers for data handling.

Calibration Range: 0.0 to .060 mg/L

Resolution: 0.001 Abs. Units

Sensitivity: 0.010 mg/L gives .210 Abs. Units

Instrument Detection Limit: 0.0002 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.0 to 0.060 mg/L

Accuracy-

Precision of Controls-

| | A | B |
|-----------|-----------|-------|
| mean | .0032mg/L | .035 |
| std. dev. | .0003mg/L | .0017 |
| R.S.D. | 8.1 % | 4.9 % |

Precision of Duplicates-low range mid range high range

s.d. .0002

mean .004

W .0002mg/L

T .0020mg/L

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

MANGANESE-GF IN PRECIPITATION

Operating Range = .00020 to 0.060 mg/L

IN - RUN DUPLICATES

| Range | <.00020 | .00020 to 0.0120 | 0.0120 to 0.0300 | 0.0300 to 0.060 | > 0.060 |
|-------|---------|------------------|------------------|-----------------|---------|
| no. | 15 | 33 | 0 | 0 | 0 |
| s.w. | | 0.00020 | 0.00000 | 0.00000 | |
| mean | | 0.0040 | 0.0000 | 0.0000 | |

QA CONTROL SAMPLES

| SAMPLE I.D. | NO. | MEAN | STD. DEV. | R.S.D. |
|-------------|-----|---------|-----------|--------|
| epal | 24 | 0.00320 | 0.00026 | 8.12 |
| epah | 24 | 0.0354 | 0.00174 | 4.92 |
| std 4 | 23 | 0.0106 | 0.00139 | 13.11 |
| std 5 | 0 | 0.0000 | 0.00000 | 0.00 |

BLANKS

| BLANK I.D. | NO. | MEAN | STD. DEV. |
|------------|-----|--------|-----------|
| blk | 0 | .00000 | .00000 |

DATE 87/02/25

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Nickel
UNIT: Water

TEST CODE: NIPDT SAMPLE TYPE: Precipdep tot
SUPERVISOR: P. Vijan

METHOD CODE: 520AF2
REVISION NO: Original
NATURE OF LAST REVISION:

DATE: 1982

SAMPLE HANDLING:

Quantity Required- 100 ml
Container- Polyethylene bag (approx. 10x20 cm) with plastic screw top
Preservative- Conc HNO₃ (0.25%) - ultra-pure
Other- Sample kept refrigerated until preserved

SAMPLE PREPARATION: Partial Extn.- Total Extn.-Yes % Extracted-

Procedure- Transfer 50 ml sample from sample bag to calibrated polyethylene tube and preserve with 1% nitric acid.
Reduce to less than 5 ml by evaporation in an oven and dilute to exactly the 5 ml mark at room temperature.
Two filtered composites, one spiked at a higher level, are also taken through this preconcentration procedure, as controls.
A typical run consists of 40 test tubes including blanks, controls, digested standards and samples.
Ni in the solutions is determined as part of a multi-element measurement system.

INTERFERENCES: None

REPORTING RESULTS: To 3 decimals

INSTRUMENTATION: Jarell Ash Atom-Comp -ICP/ES Model 975, 0.75 m, with autosampler and DEC computer for simultaneous concentration printout. PET interface for data message, storage & transfer to computer

Calibration Range: 0 to 100 µg/ml

Resolution:

Sensitivity:

Instrument Detection Limit: 0.03 µg/ml

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0 to 1 µg/ml

Accuracy-109% at 0.225 µg/ml

Precision of Controls-

A

B

mean

std. dev.

R.S.D.

Precision of Duplicates-low range

mid range

high range

s.d. 0.00007

0.00026

0.00035

mean

W

T

CONTROL LIMITS:

REMARKS: Occasional use of graphite furnace AAS is permitted as backup instrument in event of equipment failure, specific problem solving and to expedite analyses if fewer than four metals are to be determined.

SUMMARY REPORT OF QUALITY CONTROL DATA

NICKEL IN PRECIPITATION

Operating Range = .00010 to 0.010 mg/L

IN - RUN DUPLICATES

| | | | | | |
|-------|---------|------------------|------------------|-----------------|---------|
| Range | <.00010 | .00010 to 0.0020 | 0.0020 to 0.0050 | 0.0050 to 0.010 | > 0.010 |
| no. | 44 | 1 | 0 | 1 | 1 |
| s.w. | | 0.00110 | 0.00000 | 0.00040 | |
| mean | | 0.0007 | 0.0000 | 0.0066 | |

QA CONTROL SAMPLES

| SAMPLE I.D. | NO. | MEAN | STD. DEV. | R.S.D. |
|-------------|-----|---------|-----------|--------|
| std | 24 | 0.00300 | 0.00071 | 23.67 |
| qcstd | 17 | 0.2194 | 0.02425 | 11.05 |

BLANKS

| BLANK I.D. | NO. | MEAN | STD. DEV. |
|------------|-----|--------|-----------|
| BLK | 0 | .00000 | .00000 |

DATE 87/08/07

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Nickel TEST CODE: NIPDT SAMPLE TYPE: Precipdep tot
UNIT: Water SUPERVISOR: P. Vijan

METHOD CODE: 520AF2
REVISION NO: Original DATE: 1982
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 100 ml
Container- Polyethylene bag (approx. 10x20 cm) with plastic screw top
Preservative- Conc HNO₃ (0.25%) - ultra-pure
Other- Sample kept refrigerated until preserved

SAMPLE PREPARATION: Partial Extn.- Total Extn.-Yes % Extracted-
Procedure- Transfer 50 ml sample from sample bag to calibrated
polyethylene test tube and preserve with 1% nitric acid.
Analyze by graphite furnace a.a.s. with 90 µL of sample, an
argon internal flow of 0 lpm, and in peak height mode. The other
furnace conditions are set according to manual specifications.

INTERFERENCES: None

REPORTING RESULTS: To 4 decimals
INSTRUMENTATION: Perkin-Elmer Models 2380 or 603 AAS with PE 400 or
PE 500 graphite furnace atomizer systems, and AS 40 autosamplers. Both
interfaced with PET computers for data handling.

Calibration Range: 0.0 to .030 mg/L
Resolution: 0.001 Abs. Units
Sensitivity: 0.010 mg/L = .150 Abs. Units
Instrument Detection Limit: 0.0002 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.0 to 0.030 mg/L

Accuracy-

Precision of Controls-

| | A | B |
|-----------|-----------|-------|
| mean | .0022mg/L | .0207 |
| std. dev. | .0002mg/L | .0012 |
| R.S.D. | 7.3 % | 5.9 % |

Precision of Duplicates-low range

mid range

high range

s.d. 0.0001 0.0001

mean 0.0008 0.0077

W .001 mg/L

T .005 mg/L

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

NICKEL-GF IN PRECIPITATION

Operating Range = .00020 to 0.030 mg/L

IN - RUN DUPLICATES

| Range | <.00020 | .00020 to 0.0060 | 0.0060 to 0.0150 | 0.0150 to 0.030 | > 0.030 |
|-------|---------|------------------|------------------|-----------------|---------|
| no. | 32 | 18 | 2 | 0 | 3 |
| s.w. | | 0.00010 | 0.00010 | 0.00000 | |
| mean | | 0.0008 | 0.0077 | 0.0000 | |

QA CONTROL SAMPLES

| SAMPLE I.D. | NO. | MEAN | STD. DEV. | R.S.D. |
|-------------|-----|---------|-----------|--------|
| epal | 27 | 0.00220 | 0.00016 | 7.27 |
| epah | 16 | 0.0207 | 0.00123 | 5.94 |
| std 4 | 25 | 0.0102 | 0.00129 | 12.65 |
| std 5 | 0 | 0.0000 | 0.00000 | 0.00 |

BLANKS

| BLANK I.D. | NO. | MEAN | STD. DEV. |
|------------|-----|--------|-----------|
| blk | 0 | .00000 | .00000 |

DATE 87/02/25

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Vanadium TEST CODE: VVPDT SAMPLE TYPE: Precipdep tot
UNIT: Water SUPERVISOR: P. Vijan

METHOD CODE: 520AF2
REVISION NO: Original DATE: 1982
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 100 ml
Container- Polyethylene bag (approx. 10x20 cm) with plastic screw top
Preservative- Conc HNO₃ (0.25%) - ultra-pure
Other- Sample kept refrigerated until preserved

SAMPLE PREPARATION: Partial Extn.- Total Extn.-Yes % Extracted-
Procedure- Transfer 50 ml sample from sample bag to calibrated
polyethhylene tube and preserve with 1% nitric acid.
Reduce to less than 5 ml by evaporation in an oven and dilute to
exactly the 5 ml mark at room temperature.
Two filtered composites, one spiked at a higher level, are also
taken through this preconcentration procedure, as controls.
A typical run consists of 40 test tubes including blanks, controls,
digested standards and samples.
V in the solutions is determined as part of a multielement
measurement system.

INTERFERENCES: None

REPORTING RESULTS: To 3 decimals

INSTRUMENTATION: Jarell Ash Atom-Comp -ICP/ES Model 975, 0.75 m,
with autosampler and DEC computer for simultaneous concentration
printout. PET interface for data message, storage & transfer to computer

Calibration Range: 0 to 100 µg/ml

Resolution:

Sensitivity:

Instrument Detection Limit: 0.008 µg/ml

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0 to ? µg/ml

Accuracy-101% at 0.86 µg/ml

Precision of Controls-

A

B

mean

std. dev.

R.S.D.

Precision of Duplicates-low range

mid range

high range

s.d. 0.00004

0.00013

0.00047

mean

W

T

CONTROL LIMITS:

REMARKS: Occasional use of graphite furnace AAS is permitted as
backup instrument in event of equipment failure, specific problem
solving and to expedite analyses if fewer than four metals are to be
determined.

SUMMARY REPORT OF QUALITY CONTROL DATA

VANADIUM IN PRECIPITATION

Operating Range = .00020 to 0.050 mg/L

IN - RUN DUPLICATES

| Range | <.00020 | .00020 to 0.0100 | 0.0100 to 0.0250 | 0.0250 to 0.050 | > 0.050 |
|-------|---------|------------------|------------------|-----------------|---------|
| no. | 33 | 14 | 0 | 0 | 0 |
| s.w. | | 0.00040 | 0.00000 | 0.00000 | |
| mean | | 0.0004 | 0.0000 | 0.0000 | |

QA CONTROL SAMPLES

| SAMPLE I.D. | NO. | MEAN | STD. DEV. | R.S.D. |
|-------------|-----|---------|-----------|--------|
| std | 25 | 0.00280 | 0.00086 | 30.71 |
| qcstd | 15 | 0.2105 | 0.04110 | 19.52 |

BLANKS

| BLANK I.D. | NO. | MEAN | STD. DEV. |
|------------|-----|--------|-----------|
| BLK | 3 | .02510 | .02171 |

DATE 87/08/07

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Vanadium TEST CODE: VVPDT SAMPLE TYPE: Precipdep tot
UNIT: Water SUPERVISOR: P. Vijan

METHOD CODE: 520AF2

REVISION NO: Original

DATE: 1982

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 100 ml

Container- Polyethylene bag (approx. 10x20 cm) with plastic screw top

Preservative- Conc HNO₃ (0.25%) - ultra-pure

Other- Sample kept refrigerated until preserved

SAMPLE PREPARATION: Partial Extn.- Total Extn.-Yes % Extracted-

Procedure- Transfer 50 ml sample from sample bag to calibrated polyethylene test tube and preserve with 1% nitric acid.

Analyze by graphite furnace a.a.s. with 90 µL of sample, an argon internal flow of 0 lpm, and in peak height mode. The other furnace conditions are set according to manual specifications.

INTERFERENCES: None

REPORTING RESULTS: To 4 decimals

INSTRUMENTATION: Perkin-Elmer Models 2380 or 603 AAS with PE 400 or PE 500 graphite furnace atomizer systems, and AS 40 autosamplers. Both interfaced with PET computers for data handling.

Calibration Range: 0.0 to .010 mg/L

Resolution: 0.001 Abs. Units

Sensitivity: 0.002 mg/L = 0.018 Abs. Units

Instrument Detection Limit: 0.0004 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.0 to 0.010 mg/L

Accuracy-

Precision of Controls-

| | A | B |
|-----------|-----------|--------|
| mean | .0085mg/L | .010 |
| std. dev. | .0020mg/L | .0012 |
| R.S.D. | 23.4 % | 12.3 % |

Precision of Duplicates-low range

mid range

high range

| | | |
|------|-------|-------|
| s.d. | .0001 | .0006 |
| mean | .001 | .002 |

W .0001mg/L

T .0010mg/L

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

VANADIUM-GF IN PRECIPITATION

Operating Range = .00010 to 0.010 mg/L

IN - RUN DUPLICATES

| Range | <.00010 | .00010 to 0.0020 | 0.0020 to 0.0050 | 0.0050 to 0.010 | > 0.010 |
|-------|---------|------------------|------------------|-----------------|---------|
| no. | 46 | 7 | 1 | 0 | 0 |
| s.w. | | 0.00010 | 0.00060 | 0.00000 | |
| mean | | 0.0010 | 0.0020 | 0.0000 | |

QA CONTROL SAMPLES

| SAMPLE I.D. | NO. | MEAN | STD. DEV. | R.S.D. |
|-------------|-----|---------|-----------|--------|
| epal | 25 | 0.00850 | 0.00199 | 23.41 |
| epah | 0 | 0.0000 | 0.00000 | 0.00 |
| std 4 | 18 | 0.0101 | 0.00124 | 12.28 |
| std 5 | 0 | 0.0000 | 0.00000 | 0.00 |

BLANKS

| BLANK I.D. | NO. | MEAN | STD. DEV. |
|------------|-----|--------|-----------|
| blk | 0 | .00000 | .00000 |

DATE 87/02/25

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Zinc TEST CODE: ZNPDT SAMPLE TYPE: Precipdep tot
UNIT: Water SUPERVISOR: P. Vijan

METHOD CODE: 520AF2
REVISION NO: Original DATE: 1982
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 100 ml
Container- Polyethylene bag (approx. 10x20 cm) with plastic screw top
Preservative- Conc HNO₃ (0.25%) - ultra-pure
Other- Sample kept refrigerated until preserved

SAMPLE PREPARATION: Partial Extn.- Total Extn.-Yes % Extracted-
Procedure- Transfer 50 ml sample from sample bag to calibrated
polyethhylene tube and preserve with 1% nitric acid.
Reduce to less than 5 ml by evaporation in an oven and dilute to
exactly the 5 ml mark at room temperature.
Two filtered composites, one spiked at a higher level, are also
taken through this preconcentration procedure, as controls.
A typical run consists of 40 test tubes including blanks, controls,
digested standards and samples.
Zn in the solutions is determined as part of a multielement
measurement system.

INTERFERENCES: None

REPORTING RESULTS: To 3 decimals

INSTRUMENTATION: Jarell Ash Atom-Comp -ICP/ES Model 975, 0.75 m,
with autosampler and DEC computer for simultaneous concentration
printout. PET interface for data message, storage & transfer to computer
Calibration Range: 0 to 100 µg/ml

Resolution:

Sensitivity:

Instrument Detection Limit: 0.005 µg/ml

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0 to 2 µg/ml

Accuracy-108% at 0.45 µg/ml

Precision of Controls-

A

B

mean

std. dev.

R.S.D.

Precision of Duplicates-low range

mid range

high range

s.d. 0.0004

0.0009

0.0014

mean

W

T

CONTROL LIMITS:

REMARKS: Occasional use of graphite furnace AAS is permitted as
backup instrument in event of equipment failure, specific problem
solving and to expedite analyses if fewer than four metals are to be
determined.

SUMMARY REPORT OF QUALITY CONTROL DATA

ZINC

IN PRECIPITATION

Operating Range = .00050 to 0.020 mg/L

IN - RUN DUPLICATES

| | | | | | |
|-------|---------|------------------|------------------|-----------------|---------|
| Range | <.00050 | .00050 to 0.0040 | 0.0040 to 0.0100 | 0.0100 to 0.020 | > 0.020 |
| no. | 3 | 13 | 27 | 2 | 2 |
| s.w. | | 0.00140 | 0.00030 | 0.00070 | |
| mean | | 0.0027 | 0.0057 | 0.0117 | |

QA CONTROL SAMPLES

| SAMPLE I.D. | NO. | MEAN | STD. DEV. | R.S.D. |
|-------------|-----|---------|-----------|--------|
| std | 28 | 0.00370 | 0.00315 | 85.14 |
| qcstd | 16 | 0.2420 | 0.03032 | 12.53 |

BLANKS

| BLANK I.D. | NO. | MEAN | STD. DEV. |
|------------|-----|--------|-----------|
| BLK | 21 | .12250 | .23362 |

DATE 87/08/07

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Zinc
UNIT: Water

TEST CODE: ZNPDT SAMPLE TYPE: Precipdep tot
SUPERVISOR: P. Vijan

METHOD CODE: 520AF2

REVISION NO: Original

DATE: 1982

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 100 ml

Container- Polyethylene bag (approx. 10x20 cm) with plastic screw top

Preservative- Conc HNO₃ (0.25%) - ultra-pure

Other- Sample kept refrigerated until preserved

SAMPLE PREPARATION: Partial Extn.- Total Extn.-Yes % Extracted-

Procedure- Transfer 50 ml sample from sample bag to calibrated polyethylene test tube and preserve with 1% nitric acid.

Analyze by graphite furnace a.a.s. with 20 µL of sample, an argon internal flow of 50 lpm, and in peak height mode. The other furnace conditions are set according to manual specifications.

INTERFERENCES: None

REPORTING RESULTS: To 3 decimals

INSTRUMENTATION: Perkin-Elmer Models 2380 or 603 AAS with PE 400 or PE 500 graphite furnace atomizer systems, and AS 40 autosamplers. Both interfaced with PET computers for data handling.

Calibration Range: 0.0 to .100 mg/L

Resolution: 0.001 Abs. Units

Sensitivity: 0.0300 mg/L gives .600 Abs. Units

Instrument Detection Limit: 0.001 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.0 to 0.100 mg/L

Accuracy-

Precision of Controls-

| | A | B |
|-----------|-----------|-------|
| mean | .004 mg/L | .041 |
| std. dev. | .0005mg/L | .0027 |
| R.S.D. | 11.8 % | 6.5 % |

Precision of Duplicates-low range mid range high range

s.d. .0004 0.0088

mean .004 0.049

W .001 mg/L

T .005 mg/L

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

ZINC-GF

IN PRECIPITATION

Operating Range = .00100 to 0.100 mg/L

IN - RUN DUPLICATES

| Range | <.00100 | .00100 to 0.0200 | 0.0200 to 0.0500 | 0.0500 to 0.100 | > 0.100 |
|-------|---------|------------------|------------------|-----------------|---------|
| no. | 18 | 27 | 1 | 0 | 0 |
| s.w. | | 0.00040 | 0.00880 | 0.00000 | |
| mean | | 0.0040 | 0.0490 | 0.0000 | |

QA CONTROL SAMPLES

| SAMPLE I.D. | NO. | MEAN | STD. DEV. | R.S.D. |
|-------------|-----|---------|-----------|--------|
| epal | 26 | 0.00400 | 0.00047 | 11.75 |
| epah | 21 | 0.0411 | 0.00267 | 6.50 |
| std 4 | 23 | 0.0110 | 0.00316 | 28.73 |
| std 5 | 22 | 0.0219 | 0.00264 | 12.05 |

BLANKS

| BLANK I.D. | NO. | MEAN | STD. DEV. |
|------------|-----|--------|-----------|
| blk | 11 | .00250 | .00109 |

DATE 87/02/25

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Aluminum TEST CODE:ALPBT SAMPLE TYPE:PrecipNL
UNIT: Water SUPERVISOR: P. Vijan

METHOD CODE:521AF2
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- Approx. 20 ml
Container-Polyethylene bag (approx. 35x20 cm) with plastic screw top
Preservative- Nitric acid (5% v/v) - A.R grade.
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted-
Procedure- Leach sample bags for 24 h with 1000 ml 5% HNO3 and
save for analysis in polyethylene centrifuge tubes.
Transfer to sample cups of autosampler and determine Al by graphite
AAS as part of a multielement analytical system.
Forty samples, including blanks, standards and controls, constitute
a typical batch for the measurement step. The raw data is converted
into printed analytical results through a PET computer.
Set the instrument so that 20 µL of sample is delivered into furnace
and the internal argon flow is 50 lpm. The signal is measured in the
peak height mode.

INTERFERENCES: None

REPORTING RESULTS: To 3 decimals

INSTRUMENTATION: Perkin-Elmer Models 603 and 2380 AAS with
P-E 500 and P-E 400 graphite furnace atomizer systems, respectively,
and AS-40 autosamplers. Both interfaced with PET for data handling.

Calibration Range:0.001 to 0.100 mg/L

Resolution: 0.001 abs.

Sensitivity:0.030 mg/L = .200 Abs. Units

Instrument Detection Limit: 0.005 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.0 to 0.100 mg/L

Accuracy-105% at 0.0126 µg/ml; 94% at 0.079 µg/ml

Precision of Controls-

| | A | B |
|-----------|-----------|-------|
| mean | .007 mg/L | 0.080 |
| std. dev. | .0015mg/L | 0.006 |
| R.S.D. | 23.2 % | 7.5 % |

| Precision of Duplicates- | low range | mid range | high range |
|--------------------------|-----------|-----------|------------|
| s.d. | 0.0010 | 0.0019 | 0.0006 |
| mean | 0.009 | 0.028 | 0.061 |

W .001 mg/L

T .010 mg/L

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

ALUMINUM-GF IN NLPRECIP

Operating Range = .00100to 0.100 mg/L

IN - RUN DUPLICATES

Range <.00100 .00100to0.0200 0.0200to0.0500 0.0500to 0.100 > 0.100

| | | | | | |
|------|---|---------|---------|---------|---|
| no. | 1 | 21 | 17 | 3 | 1 |
| s.w. | | 0.00100 | 0.00190 | 0.00060 | |
| mean | | 0.0090 | 0.0280 | 0.0610 | |

QA CONTROL SAMPLES

| SAMPLE I.D. | NO. | MEAN | STD. DEV. | R.S.D. |
|-------------|-----|---------|-----------|--------|
| epal | 27 | 0.00650 | 0.00151 | 23.23 |
| epah | 29 | 0.0801 | 0.00597 | 7.45 |
| std 4 | 28 | 0.0107 | 0.00263 | 24.58 |
| std 5 | 23 | 0.0215 | 0.00433 | 20.14 |

BLANKS

| BLANK I.D. | NO. | MEAN | STD. DEV. |
|------------|-----|--------|-----------|
| blk | 17 | .00630 | .00113 |

DATE 87/02/25

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Copper TEST CODE: CUPBT SAMPLE TYPE: PrecipNL
UNIT: Water SUPERVISOR: P Vijan

METHOD CODE: 521AF2
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- Approx. 20 ml
Container- Polyethylene bag (approx. 35x20 cm) with plastic screw top
Preservative- Nitric acid (5% v/v) - A.R grade.
Other-

SAMPLE PREPARATION: Partial Extn.- Total Extn.-Yes % Extracted-
Procedure- Leach sample bags for 24 h with 1000 ml 5% HNO₃ and
save for analysis in polyethylene centrifuge tubes.
Transfer to sample cups of autosampler and determine Cu by graphite
AAS as part of a multielement analytical system.
Forty samples, including blanks, standards and controls, constitute
a typical batch for the measurement step. The raw data is converted
into printed analytical results through a PET computer.
Set the instrument so that 90 µL of sample is delivered into furnace
and the internal argon flow is 50 lpm. The signal is measured in the
peak height mode.

INTERFERENCES: None

REPORTING RESULTS: 4 significant figures
INSTRUMENTATION: Perkin-Elmer Models 603 and 2380 AAS with
P-E 500 and P-E 400 graphite furnace atomizer systems, respectively,
and AS-40 autosamplers. Both interfaced with PET for data handling.

Calibration Range: 0.0002 to 0.060 mg/L
Resolution: 0.001 abs.
Sensitivity: 0.030 mg/L = 0.250 Abs. Units
Instrument Detection Limit: 0.0002 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.0 to 0.060 mg/L

Accuracy- Data to be updated

Precision of Controls-

| | A | B |
|-----------|-----------|--------|
| mean | .0034mg/L | 0.0328 |
| std. dev. | .0002mg/L | 0.0017 |
| R.S.D. | 6.5 % | 5.2 % |

Precision of Duplicates-low range mid range high range

| | | |
|------|--------|--------|
| s.d. | 0.0002 | 0.0007 |
| mean | 0.0009 | 0.0235 |

W .0001mg/L T .0010mg/L

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

COPPER-GF IN NLPRECIP

Operating Range = .00010 to 0.060 mg/L

IN - RUN DUPLICATES

| Range | <.00010 | .00010 to 0.0120 | 0.0120 to 0.0300 | 0.0300 to 0.060 | > 0.060 |
|-------|---------|------------------|------------------|-----------------|---------|
| no. | 27 | 17 | 1 | 0 | 0 |
| s.w. | | 0.00015 | 0.00071 | 0.00000 | |
| mean | | 0.0009 | 0.0235 | 0.0000 | |

QA CONTROL SAMPLES

| SAMPLE I.D. | NO. | MEAN | STD. DEV. | R.S.D. |
|-------------|-----|---------|-----------|--------|
| epal | 30 | 0.00340 | 0.00022 | 6.47 |
| epah | 12 | 0.0328 | 0.00171 | 5.21 |
| std 4 | 22 | 0.0099 | 0.00120 | 12.12 |
| std 5 | 15 | 0.0209 | 0.00218 | 10.43 |

BLANKS

| BLANK I.D. | NO. | MEAN | STD. DEV. |
|------------|-----|--------|-----------|
| blk | 10 | .00020 | .00004 |

DATE 87/02/25

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Iron TEST CODE: FEPBT SAMPLE TYPE: PrecipNL
UNIT: Water SUPERVISOR: P Vijan

METHOD CODE: 521AF2
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- Approx. 20 ml
Container- Polyethylene bag (approx. 35x20 cm) with plastic screw top
Preservative- Nitric acid (5% v/v) - A.R grade.
Other-

SAMPLE PREPARATION: Partial Extn.- Total Extn.-Yes % Extracted-
Procedure- Leach sample bags for 24 h with 1000 ml 5% HNO₃ and
save for analysis in polyethylene centrifuge tubes.
Transfer to sample cups of autosampler and determine Fe by graphite
AAS as part of a multielement analytical system.
Forty samples, including blanks, standards and controls, constitute
a typical batch for the measurement step. The raw data is converted
into printed analytical results through a PET computer.
Set the instrument so that 20 µL of sample is delivered into furnace
and the internal argon flow is 50 lpm. The signal is measured in the
peak height mode.

INTERFERENCES: None

REPORTING RESULTS: To 3 decimals

INSTRUMENTATION: Perkin-Elmer Models 603 and 2380 AAS with
P-E 500 and P-E 400 graphite furnace atomizer systems, respectively,
and AS-40 autosamplers. Both interfaced with PET for data handling.

Calibration Range: 0.001 to 0.1 mg/L

Resolution: 0.001 abs.

Sensitivity: 0.030 = .200 Abs. Units

Instrument Detection Limit: 0.001 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.0 to 0.100 mg/L

Accuracy- 110% at 0.0044 µg/ml: 108% at 0.0215 µg/ml

Precision of Controls-

| | A | B |
|-----------|-----------|-------|
| mean | .008 mg/L | 0.080 |
| std. dev. | .0009mg/L | 0.004 |
| R.S.D. | 11.2 % | 5.0 % |

| Precision of Duplicates- | low range | mid range | high range |
|--------------------------|-----------|-----------|------------|
| s.d. | 0.0018 | 0.0019 | 0.0015 |
| mean | 0.013 | 0.033 | 0.058 |

W .0005mg/L

T .0050mg/L

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

IRON-GF

IN NLPRECIP

Operating Range = .00100to 0.100 mg/L

IN - RUN DUPLICATES

| | | | | | |
|-------|---------|----------------|----------------|----------------|---------|
| Range | <.00100 | .00100to0.0200 | 0.0200to0.0500 | 0.0500to 0.100 | > 0.100 |
| no. | 3 | 20 | 18 | 3 | 3 |
| s.w. | | 0.00180 | 0.00190 | 0.00150 | |
| mean | | 0.0130 | 0.0330 | 0.0580 | |

QA CONTROL SAMPLES

| SAMPLE I.D. | NO. | MEAN | STD. DEV. | R.S.D. |
|-------------|-----|---------|-----------|--------|
| epal | 30 | 0.00760 | 0.00085 | 11.18 |
| epah | 27 | 0.0803 | 0.00401 | 4.99 |
| std 4 | 30 | 0.0110 | 0.00192 | 17.45 |
| std 5 | 26 | 0.0231 | 0.00249 | 10.78 |

BLANKS

| BLANK I.D. | NO. | MEAN | STD. DEV. |
|------------|-----|--------|-----------|
| blk | 46 | .00180 | .00076 |

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Lead TEST CODE: PBPBT SAMPLE TYPE: PrecipNL
UNIT: Water SUPERVISOR: P. Vijan

METHOD CODE: 521AF2
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- Approx. 20 ml
Container- Polyethylene bag (approx. 35x20 cm) with plastic screw top
Preservative- Nitric acid (5% v/v) - A.R grade.
Other-

SAMPLE PREPARATION: Partial Extn.- Total Extn.-Yes % Extracted-

Procedure- Leach sample bags for 24 h with 1000 ml 5% HNO₃ and
save for analysis in polyethylene centrifuge tubes.
Transfer to sample cups of autosampler and determine Pb by graphite
AAS as part of a multielement analytical system.
Forty samples, including blanks, standards and controls, constitute
a typical batch for the measurement step. The raw data is converted
into printed analytical results through a PET computer.
Set the instrument so that 20 µL of sample is delivered into furnace
and the internal argon flow is 50 lpm. The signal is measured in the
peak height mode.

INTERFERENCES:-None

REPORTING RESULTS: To 3 decimals

INSTRUMENTATION: Perkin-Elmer Models 603 and 2380 AAS with
P-E 500 and P-E 400 graphite furnace atomizer systems, respectively,
and AS-40 autosamplers. Both interfaced with PET for data handling.

Calibration Range: 0.001 to 0.100 mg/L

Resolution: 0.001 abs.

Sensitivity: 0.030 mg/L = .200 Abs. Units

Instrument Detection Limit: 0.001 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.0 to 0.100 mg/L

Accuracy- 94% at 0.0045 µg/ml; 84% at 0.020 µg/ml

Precision of Controls-

| | A | B |
|-----------|-----------|--------|
| mean | .0044mg/L | 0.0419 |
| std. dev. | .0003mg/L | 0.0084 |
| R.S.D. | 6.4 % | 20.1 % |

Precision of Duplicates-low range mid range high range

s.d. 0.0006

mean 0.003

W .001 mg/L

T .010 mg/L

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

LEAD-GF

IN NLPRECIP

Operating Range = .00100to 0.100 mg/L

IN - RUN DUPLICATES

| | | | | | |
|-------|---------|----------------|----------------|----------------|---------|
| Range | <.00100 | .00100to0.0200 | 0.0200to0.0500 | 0.0500to 0.100 | > 0.100 |
| no. | 25 | 18 | 0 | 1 | 0 |
| s.w. | | 0.00060 | 0.00000 | 0.00640 | |
| mean | | 0.0030 | 0.0000 | 0.0950 | |

QA CONTROL SAMPLES

| SAMPLE I.D. | NO. | MEAN | STD. DEV. | R.S.D. |
|-------------|-----|---------|-----------|--------|
| epal | 27 | 0.00440 | 0.00028 | 6.36 |
| epah | 26 | 0.0419 | 0.00844 | 20.14 |
| std 4 | 21 | 0.0112 | 0.00217 | 19.37 |
| std 5 | 15 | 0.0230 | 0.00286 | 12.43 |

BLANKS

| BLANK I.D. | NO. | MEAN | STD. DEV. |
|------------|-----|--------|-----------|
| blk | 27 | .00260 | .00114 |

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Zinc TEST CODE: ZNPBT SAMPLE TYPE: PrecipNL
UNIT: Water SUPERVISOR: P. Vijan

METHOD CODE: 521AF2
REVISION NO: Original DATE: 1980
NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- Approx. 20 ml
Container- Polyethylene bag (approx. 35x20 cm) with plastic screw top
Preservative- Nitric acid (5% v/v) - A.R grade.
Other-

SAMPLE PREPARATION: Partial Extn.- Total Extn.-Yes % Extracted-

Procedure- Leach sample bags for 24 h with 1000 ml 5% HNO₃ and
save for analysis in polyethylene centrifuge tubes.
Transfer to sample cups of autosampler and determine Zn by graphite
AAS as part of a multielement analytical system.
Forty samples, including blanks, standards and controls, constitute
a typical batch for the measurement step. The raw data is converted
into printed analytical results through a PET computer.
Set the instrument so that 20 µL of sample is delivered into furnace
and the internal argon flow is 50 lpm. The signal is measured in the
peak height mode.

INTERFERENCES: None

REPORTING RESULTS: 3 significant figures

INSTRUMENTATION: Perkin-Elmer Models 603 and 2380 AAS with
P-E 500 and P-E 400 graphite furnace atomizer systems, respectively,
and AS-40 autosamplers. Both interfaced with PET for data handling.

Calibration Range: 0.001 to 0.100 mg/L

Resolution: 0.001 abs.

Sensitivity: 0.030 mg/L = .600 Abs. Units

Instrument Detection Limit: 0.001 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.0 to 0.060 mg/L

Accuracy- Data to be updated

Precision of Controls-

| | A | B |
|-----------|-----------|-------|
| mean | .004 mg/L | 0.040 |
| std. dev. | .0003mg/L | 0.003 |
| R.S.D. | 7.8 % | 8.0 % |

| Precision of Duplicates- | low range | mid range | high range |
|--------------------------|-----------|-----------|------------|
| s.d. | 0.0006 | 0.0011 | 0.0014 |
| mean | 0.005 | 0.031 | 0.040 |

W .0005mg/L

T .0050mg/L

CONTROL LIMITS:

REMARKS:

1.67

SUMMARY REPORT OF QUALITY CONTROL DATA

ZINC-GF

IN NLPRECIP

Operating Range = .00100to 0.100 mg/L

IN - RUN DUPLICATES

| | | | | | |
|-------|---------|----------------|----------------|----------------|---------|
| Range | <.00100 | .00100to0.0200 | 0.0200to0.0500 | 0.0500to 0.100 | > 0.100 |
| no. | 19 | 26 | 2 | 0 | 0 |
| s.w. | | 0.00060 | 0.00110 | 0.00000 | |
| mean | | 0.0050 | 0.0310 | 0.0000 | |

QA CONTROL SAMPLES

| SAMPLE I.D. | NO. | MEAN | STD. DEV. | R.S.D. |
|-------------|-----|---------|-----------|--------|
| epal | 29 | 0.00400 | 0.00031 | 7.75 |
| epah | 24 | 0.0404 | 0.00321 | 7.95 |
| std 4 | 27 | 0.0109 | 0.00221 | 20.28 |
| std 5 | 22 | 0.0219 | 0.00264 | 12.05 |

BLANKS

| BLANK I.D. | NO. | MEAN | STD. DEV. |
|------------|-----|--------|-----------|
| blk | 11 | .00250 | .00109 |



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MOE/ANN/ITC/ALRJ

MOE/ANN/ITC/ALRJ
Ontario Ministry of the En
Annual quality
assurance alrj
c.1 a aa